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May 1, 1996

Via Telecopy and U.S. Mail

Tim Murphy  
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RECEIVED

MAY 03 1996

IEPA/DLPC

Re: Carus Chemical Company

Dear Tim:

Concerning the status of the Carus Chemical matter, we have received and reviewed the draft letter you forwarded to us in late March. I have tried calling on various occasions to discuss our comments to the letter. (I assume you have been busy with spring field work!) Please call me at your earliest convenience to discuss any necessary changes to the letter. Thank you. In the meantime, Carus has been in the process of preparing a work plan for the Phase III (Muddies) area and a proposed schedule for overall work at the various sites.

Very truly yours,

MAUCK, BELLANDE & CHEELY

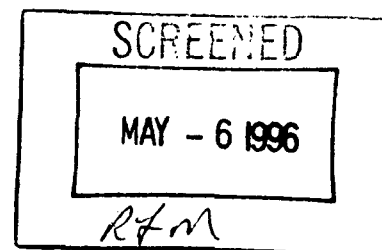
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MRS:lm1

cc: Roger Threde

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**GEOSYNTEC CONSULTANTS**

0998160003  
Carus Chemical Co.  
SF Tech

28 February 1994

Mr. Tim Murphy  
Remedial Project Management Section, Bureau of Land  
Illinois Environmental Protection Agency  
2200 Churchill Road  
Springfield, Illinois 62794

Subject: Proposed Work Plan for Phase II Site Investigation  
Carus Chemical Company, LaSalle, Illinois

Dear Mr. Murphy:

GeoSyntec Consultants (GeoSyntec) is pleased to submit three copies of the "Proposed Work Plan for the Phase II Site Investigation - Carus Chemical Company Manufacturing Facility, LaSalle, Illinois" (Phase II Work Plan). This Phase II Work Plan has been completed as part of a continuing site investigation initiated by the Carus Chemical Company pursuant to the Agency's Pre-Notice Site Cleanup Program.

If you have any questions or comments, please do not hesitate to contact either of the undersigned. As always, and on behalf of the Carus Chemical Company, GeoSyntec looks forward to working with you.

Sincerely,

Nandra D. Weeks, P.E.  
Project Manager

Neil D. Williams, Ph.D., P.E.  
Principal

Copies to: Roger C. Threde  
Mark R. Sargis

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**SCREENED BF**

**PROPOSED WORK PLAN FOR  
PHASE II SITE INVESTIGATION -  
CARUS CHEMICAL COMPANY  
MANUFACTURING FACILITY  
LASALLE, ILLINOIS**

Prepared for



1500 Eighth Street  
LaSalle, Illinois 61301

**RECEIVED**

MAR 02 1994

IEPA/DLPC

Prepared by



**GEOSYNTEC CONSULTANTS**

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GeoSyntec Consultants Project No. FE2167

February 1994

## WORK PLAN

1

2

3



**PROPOSED WORK PLAN FOR  
PHASE II SITE INVESTIGATION -  
CARUS CHEMICAL COMPANY  
MANUFACTURING FACILITY  
LASALLE, ILLINOIS**

Prepared for

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1500 Eighth Street  
LaSalle, Illinois 61301**

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**February 1994**

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## 1. INTRODUCTION

### 1.1 Terms of Reference

GeoSyntec Consultants (GeoSyntec) is pleased to submit this document entitled "*Proposed Work Plan for Phase II Site Investigation - Carus Chemical Company Manufacturing Facility*" (hereinafter referred to as the Work Plan) to the Illinois Environmental Protection Agency (IEPA). This document was prepared on behalf of the Carus Chemical Company, which is located in LaSalle, Illinois, and is being submitted as part of the Pre-Notice Site Cleanup Program in accordance with the Evaluation Services Agreement dated 28 June 1993. This Work Plan summarizes the planned efforts to be undertaken in the area to the east of the Carus Chemical Company main plant, which is designated as the Phase II Site Investigation area.

### 1.2 Purpose of the Phase II Work Plan

The purposes of the Phase II Work Plan are:

- to document the decisions and evaluations made during the scoping process;
- to outline the work elements to be completed during the Phase II Site Investigation;
- to provide mechanisms for technical documentation and control during the implementation of the Phase II Site Investigation; and
- to provide mechanisms for managing the Phase II Site Investigation.

The intended primary users of the Work Plan are the Carus Chemical Company, who will be responsible for overall management of the work, the IEPA, who will be the lead public agency in oversight of the work, and GeoSyntec, who will be responsible for performing the scope of services described herein.

### 1.3 Purpose and Scope of the Investigation

The purposes of the Phase II Site Investigation are to: (i) characterize the nature and extent of chemical constituents in the Phase II area of the site; (ii) determine the impact on ground water and the Little Vermilion River; and (iii) identify the potential sources of any such impact. Following completion of the investigation, this information will be used as the basis for assessing the environmental risk posed by the site and for developing and evaluating appropriate remedial alternatives.

The scope of work consists of scientific investigations of the Phase II area of the site, including:

- environmental field work, such as drilling, sampling, and hydrogeologic testing;
- laboratory analysis of ground water, surface water, soil, and sediment for constituents of concern;
- design, installation, and testing of a monitoring-well network; and
- data analysis and report preparation.

#### 1.4 Organization of the Work Plan

The remainder of the Work Plan is organized as follows:

- background information is presented in Section 2;
- the rationale behind the scope of work is presented in Section 3;
- a description of each work element is presented in Section 4; and
- the anticipated work schedule is presented in Section 5.

## **2. BACKGROUND**

### **2.1 Overview**

This section provides general background information about the Carus Chemical Company site. The remainder of the section is organized as follows:

- The location and general history of the site are described in Section 2.2;
- Previous investigations are reviewed in Section 2.3; and
- The hydrogeologic setting is described in Section 2.4.

### **2.2 Site Description and History**

#### **2.2.1 Location and General History**

The Carus Chemical Company manufacturing facility is located at 1500 8th Street, in the Northwest Quarter of Section 14 and the Northeast Quarter of Section 15 in Township 33 North, Range 1 East of the Third Principal Meridian in LaSalle County, Illinois. A location map is provided as Figure 1. Further description and history of the Carus Chemical Company site and surrounding properties are contained in the Phase I Site Completion Report.

#### **2.2.2 Physiographic and Climatic Setting**

The Carus Chemical Company manufacturing facility is located within LaSalle County, which consists of a flat upland area with an average elevation of 650 to 750 ft (200 to 230 m) (1927 North American Datum).



The facility lies at an elevation of approximately 580 ft (180 m), except near the eastern boundary of the site, where the elevation decreases to approximately 460 ft (140 m) at the boundary of the Little Vermilion River. The Little Vermilion River flows north to south for approximately 1 mi (1.6 km) from the facility where it intersects with the Illinois River. The Illinois River Valley divides LaSalle County into approximately two equal sections. The Illinois River Valley is approximately 1 mi (1.6 km) wide at the County of LaSalle, with relatively steep bluffs approximately 150 ft (46 m) high.

The temperature in the City of LaSalle varies between approximately 18°F (-7.8°C) in January to 86°F (30°C) in July. Mean annual rainfall is approximately 34 in. (860 cm). The prevailing wind is from the south with an average velocity of 11 mph (18 km/h).

## 2.3 Previous Investigations

### 2.3.1 Introduction

Previous environmental investigations which have been performed by the IEPA and GeoSyntec at the Carus Chemical Company manufacturing facility are summarized in the following documents:

- "*CERCLA Preliminary Assessment Report*", Carus Chemical Company, undated, received by Carus Chemical Company on 23 September 1991, IEPA;
- "*CERCLA Screening Site Inspection Report*", Carus Chemical Company, sent to the Carus Chemical Company on 2 December 1992, IEPA;
- "*Preliminary Site Investigation Report*", Carus Chemical Company, June 1993, GeoSyntec; and

- *"Phase I Site Investigation Completion Report"*, Carus Chemical Company Manufacturing Facility, LaSalle, Illinois, January 1994, GeoSyntec.

A summary of the first three investigations is provided in the Phase I Site Completion Report. A summary of the Phase I Site Investigation is presented below.

### 2.3.2 Phase I Site Investigation

The Phase I Site Investigation was conducted in November 1993 and the findings were presented in a report which was submitted to the IEPA in January 1994. The purposes of the Phase I investigation were as follows:

- characterize the nature and extent of "hazardous substances" as defined in Section 101(14) of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) at the site;
- provide the necessary data in order to develop cleanup objectives; and
- develop the data such that a phased approach can be utilized to manage the particular areas of concern at the site.

The objectives of the Phase I Site Investigation as reported in the *"Proposed Work Plan for a Phase I Site Investigation - Carus Chemical Company Manufacturing Facility"* were as follows:

- evaluate the constituents at three locations of the site, including concentration and extent;

- assess the data to more accurately depict the hydrogeology of the site;
- investigation and identify potential migration pathways;
- obtain the data necessary to develop an action plan; and
- satisfy requirements of the Illinois Pre-Notice Site Cleanup Program.

The principle findings of the Phase I Site Investigation are described below.

- The Phase I area is underlain at shallow depth primarily by Pennsylvanian-age shales and limestones, with minor amounts of coal. Up to 20 ft (6 m) of Pleistocene-age glacial deposits are present in the northeastern corner of the Phase I area and up to 25 ft (7.5 m) of modern fill is present in the central and eastern part of the Phase I area.
- An upland flow system is present beneath the Phase I area. The Pennsylvanian shales and limestones act together as the lower confining layer. Pleistocene fine till (dense, silty gravelly sand) also acts as a lateral confining unit on the eastern side of the area. Principal water-bearing units consist of the modern fill deposits and Pleistocene-age coarse till (well-graded sandy gravel).
- During the Phase I Site Investigation, the principal source of ground water in the Phase I area was found to be fugitive discharge from the plant sewer. The sewer has since been repaired and carries used, non-contact cooling water from the plant to the discharge system at the Little Vermilion River. The cooling water is obtained from the LaSalle Municipal Water Works.

- Ground water in the Phase I area generally flows eastward toward the Little Vermilion River. Due to the configuration of the bedrock surface and the nature of the fill and glacial deposits, it appears that the majority of the ground-water flow from the Phase I area is being channelized into a narrow area in the vicinity of Soil Boring No. G-01. An exception to this observation is flow in the vicinity of Monitoring Well No. G-04, which lies in the coarse till deposits.
- Petroleum and/or petroleum constituents were observed in the soil and ground-water samples from Monitoring Well No. G-04. The petroleum appeared to be fresh diesel fuel or light fuel oil. Petroleum constituents appear to be limited to the extreme northeast corner of the Phase I area. There appears to be a potential off-site source of fresh fuel oil or diesel fuel, and no on-site source of fresh fuel oil or diesel fuel was discovered.
- Metals were found in some soil samples primarily confined to fill areas. The impacted soil samples contained elevated levels of cadmium, copper, lead, and/or zinc; however, the Toxicity Characteristic Leaching Procedure (TCLP) results indicate that none of the samples analyzed contained detectable quantities of metals in excess of the Illinois Hazardous Waste Management Regulation (IHWMR) limits (there is no IHWMR limit set for zinc).
- The ground-water impacts of most concern at the site are: (i) dissolved lead at Monitoring Well No. G-05; and (ii) dissolved zinc at Monitoring Well No. G-106, as discussed in the Phase I Site Investigation Completion Report. GeoSyntec notes that zinc standards are not applicable to ground water within fill material (including slag). The results of the Phase I Site Investigation suggest that impacts from dissolved lead and zinc in excess of Class II standards are localized to a few areas of the site,

which probably result from the monitoring well being located in fill areas containing slag and sinter. Other metals were detected above Class II standards; however, the observed concentrations likely reflect particulates in the sample and are not an indication of dissolved metals in the ground water.

## **2.4     Hydrogeologic Setting**

### **2.4.1   Regional and Site Hydrology**

LaSalle County lies within the Illinois River Drainage Basin. The Illinois River flows across the central portion of LaSalle County in a westerly direction. Overall, LaSalle County is moderately well drained, although marshes occur near the headwaters of some upland creeks. Important tributaries of the Illinois River include the Fox River, the Vermilion River, and the Little Vermilion River, the latter of which flows north to south along the eastern property boundary of the Carus Chemical Company manufacturing facility.

The most important feature influencing surface-water flow at and near the Carus Chemical Company manufacturing facility is the Little Vermilion River. Surface runoff across the site is directed toward the holding pond. The holding pond discharges to the Little Vermilion River through a National Pollution Discharge Elimination System (NPDES) permitted discharge pipe. The easternmost edge of the slag deposits occur within the 100-year flood plain of the Little Vermilion River as designated by the United States Geological Survey (USGS).

### **2.4.2   Regional Geology**

The regional geology of north-central Illinois consists of unconsolidated Pleistocene glacial deposits overlying Paleozoic

sedimentary deposits. For the purposes of this investigation, the Paleozoic strata can be divided into lower Paleozoic deposits and upper Paleozoic deposits. The lower Paleozoic deposits consist primarily of sandstone, dolomite, and shale, and are on the order of 4,800 ft (1,440 m) thick. The upper Paleozoic deposits, which are entirely of Pennsylvanian age, consist primarily of shale, limestone, sandstone, and coal, and are on the order of 400 ft (120 m) thick. The distinction between lower and upper Paleozoic deposits is significant with respect to the LaSalle Anticline, which is the most important geological structure in the region.

The LaSalle Anticline is a sharp, southwestward-dipping flexure in the lower Paleozoic sedimentary strata. The flexure was formed after deposition of the lower Paleozoic strata but before deposition of the upper Paleozoic strata. The axis of flexure on the northwest to southeast trending anticline is located approximately 1 mi (1.6 km) to the northeast of the site. The effect of this flexure is that lower Paleozoic strata that exist at depths of 1,500 ft (450 m) an approximate distance of 1.5 mi (2.5 km) to the southwest of the site are exposed at or near the ground-surface about 1 mi (1.6 km) to the northeast of the site. This sharp flexure does not occur within the upper Paleozoic (Pennsylvanian) strata, however. The upper Paleozoic deposits merely overlap and thin against the flexure.

During the Mesozoic and early Cenozoic periods in Illinois, emergence of the Paleozoic formations resulted in widespread erosion and production of a low-relief topography. During the Pleistocene Epoch, glaciers advanced over the region, scouring out softer rocks and soils. As the ice melted, large volumes of rock and soil debris were left behind in the form of glacial drift. Glacial drift deposits range up to 600 ft (180 m) in thickness in the region.

Glacial-drift thicknesses near the site were estimated from formation descriptions provided in well construction reports, submitted by well

drillers to the Illinois Department of Public Health (IDPH). Within a 5-mi (8-km) radius of the site, drift thicknesses range from less than 10 ft (3 m) to approximately 100 ft (30 m). The average thickness of glacial deposits is approximately 40 ft (12 m) within an area bounded to the south by the Illinois River and the east by the Little Vermilion River. South of the Illinois River and west of the Vermilion River, glacial deposits average approximately 60-ft (18-m) thick. Northeast of the site, across the Little Vermilion River, glacial deposits appear to be 10-ft (3-m) thick or less. Pennsylvanian age and older formations outcrop to the east of the Little Vermilion River, as the crest of the LaSalle Anticline is approached.

#### 2.4.3 Regional Hydrogeology

Aquifers within north-central Illinois are represented by sands and gravels occurring within the glacial drift as well as permeable bedrock formations, principally sandstones and dolomites. The City of LaSalle has a wellfield approximately 0.6 mi (1.0 km) south of the Carus Chemical Company manufacturing facility, within the Illinois River Valley. The water supply wells are finished in glacial sands and gravels at depths ranging from 60 to 70 ft (18 to 21 m). The City of Peru operates a municipal wellfield approximately 2.0 mi (3.2 km) northwest of the Carus Chemical Company manufacturing facility. Water is obtained from lower Paleozoic formations at depths greater than 2,000 ft (610 m) below grade.

#### 2.4.4 Geology of the Phase II Area

The Phase II area lies within the deep and narrow valley of the Little Vermilion River. The Phase I area lies on the bluffs to the west. The width of the valley from bluff to bluff is approximately 1,000 ft (300 m), and the elevation drop from the bluffs to the river is approximately 110 ft (34 m).

The valley is cut into Pennsylvanian-age bedrock that, from the bluffs to the river, consists of about 50 ft (15 m) of shale, underlain by 50 ft (15 m) of limestone, underlain by 30 ft (9 m) of shale. Approximately 20 ft (6 m) of Pleistocene-age fine till, coarse till, and silty clay are present on the bluffs in the northwestern corner of the Phase II area. The existence of Pleistocene deposits elsewhere in the Phase II area is not known at present. Alluvial deposit of unknown thickness are known to cover the natural floor of the valley. Within the past 100 years, a portion of the valley has been filled with deposits of slag. These slag deposits are believed to have buried the alluvium and to have diverted the river to the eastern side of the valley in the central and northern portions of the Phase II area.

#### 2.4.5 Ground-Water Flow in the Phase II Area

The Phase II area is believed to be underlain by a single shallow flow system consisting of two components. In the remainder of this Work Plan, this will be referred to as the Little Vermilion Flow System. Due to the nature of the Pennsylvanian shales and limestones, GeoSyntec believes that there is no significant interconnection between the Little Vermilion Flow System and deeper water supply aquifers.

The main component of the Little Vermilion Flow System consists of the river channel, the alluvium, and the slag in the lower portions of the valley. Evidence presently available suggests a common southward-flowing gradient in these deposits with gradual convergence of flow into the river. The other component of the Little Vermilion Flow System is believed to consist of flow from the upland areas directly toward the river under relatively steep hydraulic gradients.

The evidence presented above suggests that ground water beneath the Phase II area will generally be discharged at the southern end of the Phase II area through either the channel or alluvium of the Little



Vermilion River. Monitoring of this area will provide data to evaluate if chemical constituents are leaving the Carus Chemical Company property, and, if so, to what extent.

### **3. TECHNICAL APPROACH AND RATIONALE**

#### **3.1 General Approach**

The general technical approach of this investigation is as follows:

- determine the nature of any chemical constituents that might be leaving the property;
- determine the nature of any chemical constituents that might be entering the Phase II area from outside sources;
- identify possible sources of constituents of concern within the Phase II area; and
- develop a conceptual hydrogeologic model that can be used as a basis for predicting the extent, transport, fate, and ultimate significance of any chemical constituents identified.

The fieldwork associated with the Phase II Site Investigation will be conducted in a manner consistent with protection of the health and safety of all participants. Health and safety procedures are documented in the Health and Safety Plan, Appendix A to this Work Plan.

#### **3.2 Major Issues to be Resolved**

The following major issues need to be resolved during the Phase II Site Investigation:

- the mechanism of ground-water flow from the upland flow system in the Phase I area into the lowland flow system in the Phase II area;

- the extent to which water from the Little Vermilion River contacts the slag along the river bank;
- whether or not a significant thickness of alluvium is present beneath the slag and, if so, the transmissivity of the alluvium;
- the flow rate in the Little Vermilion River system at the property boundary;
- the concentrations of chemical constituents in ground water and surface water at the downstream property boundary;
- the concentrations of chemical constituents in ground water and surface water at the upstream and upgradient property boundary in order to establish background conditions;
- the extent of chemical constituents in the ground water in the Phase II area;
- the potential sources of chemical constituents, including:
  - ground water from the Phase I area,
  - the slag deposits,
  - holding pond sediments,
  - off-site sources, and
  - natural rocks and soils.

### 3.3 Specific Approaches

In order to assess environmental risk and evaluate remedial alternatives, it will be necessary to collect site-specific data. The site-specific data needs are described below.

- The volume, extent, and character of the slag deposits will be evaluated. The slag deposits form an extensive fill aquifer system and have been identified as a possible source of chemical constituents. Data for this determination will be obtained from the following sources:
  - analysis of maps and aerial photographs;
  - field observations;
  - soil borings through the base of the slag; and
  - chemical analyses of slag samples.
- The rate and paths of ground-water flow in the Phase II area will be conceptually modelled because ground water has been identified as the medium by which mobile chemical constituents could enter the environment. Data for this conceptual model will be obtained from the following sources:
  - soil borings and detailed boring logs;
  - water-level measurements in monitoring wells;
  - laboratory and in-situ permeability testing; and
  - water-level and flow-rate measurements of surface water.
- The extent and nature of any chemical constituents in the ground water and soils of the Phase II area will be evaluated. Data for this evaluation will be based on the following:
  - chemical analysis of soil and ground-water samples; and
  - the conceptual ground-water flow model.
- The extent and nature of chemical constituents in the holding pond will be investigated. The data required for this investigation will be as follows:

- the thickness and general character of the sediments;
  - the chemical composition of the sediments;
  - the hydraulic conductivity of the sediments; and
  - the character of the underlying material.
- The quality of ground water and river water downgradient of the Carus Chemical Company property will be evaluated in order to assess whether or to what extent chemical constituents are migrating off site. Data for this assessment will be obtained by collecting and chemically analyzing samples of river water and ground water at the downgradient property boundary.
  - Upstream sources of constituents of concern will be evaluated. Data for this evaluation will be obtained from samples of ground water and river water collected at the upstream or upgradient property boundary.

### 3.4 Data Quality Objectives

The data quality objectives for this investigation are described fully in the Quality Assurance Project Plan (QAPP), which is provided as Appendix B to this Work Plan. In general, as with other investigations performed by Carus Chemical Company at this site, all data will be scientifically defensible and of high quality, suitable for use in environmental risk assessments and remedial alternatives analyses.

#### 4. WORK ELEMENTS

##### 4.1 Overview

The investigation will consist of a number of work elements. The purpose and scope of each are described below. The specific procedures that will be used in the field to implement these work elements are presented in the Field Sampling Plan (FSP), which is provided as Attachment 2 to the QAPP.

The Phase II Site Investigation will be conducted using the scientific method. Intermediate findings will be evaluated continuously and incorporated into multiple working hypotheses of site conditions. Based upon these intermediate findings and working hypotheses, the Field Director will adjust the scope of work as needed to address as directly as possible key site conditions encountered. This approach creates a dynamic investigation that produces definitive results with maximum technical effectiveness.

##### 4.2 Drilling and Piezometer Installation

Approximately 22 borings will be drilled in the Phase II area. The number and locations of borings were selected to provide aerial coverage of the Phase II area and concentrated information in areas of particular interest, such as: the upgradient and downgradient property boundaries and the former gully identified during the Phase I Site Investigation. It is anticipated that these borings will be completed as environmental piezometers. Some, however, may be plugged and abandoned, in accordance with IEPA methodology. The general purpose of these borings and piezometers is to provide data for hydrogeological and chemical characterization of subsurface conditions. The anticipated locations of the borings are shown in Figure 2 and the target zone and anticipated depth are listed in Table 1. The number and locations of the borings,

as well as the parameters listed on Table 1, may be adjusted in the field at the discretion of the Field Director on the basis of conditions encountered or anticipated.

Subsurface samples will be collected from the soil borings for three purposes: (i) visual description and classification; (ii) laboratory chemical analysis; and (iii) laboratory permeability analysis. The procedures used to collect these samples are described in the FSP. The anticipated sampling schedule for each boring is given in Table 1.

The hollow-stem auger method will be the preferred method of drilling. Driven split-spoons will be the preferred method of sample collection for visual description and chemical analysis. If conditions warrant, however, other drilling and sampling methods, such as rock coring, may be used at the discretion of the Field Director.

All downhole drilling and sampling equipment will be decontaminated prior to use in each test boring. Devices that make contact with samples collected for laboratory chemical analysis will be decontaminated before each use. The purpose of decontamination is to prevent material from one boring or sample from becoming inadvertently incorporated into other samples. The procedures for decontamination are described in the FSP.

Environmental piezometers will be constructed using nominal 2-inch diameter, Schedule 40 polyvinyl chloride (PVC) monitoring well pipe and screen. The screen lengths are anticipated to be 15 ft (5 m) where they are set across the water table and 10 ft (3 m) where they are set below the water table. The details of environmental piezometer construction and development are described fully in the FSP.

### 4.3 Hydrogeologic Testing

#### 4.3.1 Overview

Hydrogeologic testing will be performed to help determine the rate and direction of ground-water flow. Three types of hydrogeologic tests will be performed: (i) water-level measurements; (ii) slug tests; and (iii) laboratory permeability tests.

#### 4.3.2 Water-Level Measurements

The level of water in a pipe or borehole indicates the gravitational potential energy (GPE) of the ground water at that point. Because water flows from areas of higher GPE to areas of lower GPE, water-level measurements can be used as the basis for evaluating the direction of ground-water flow. Because the rate of flow is proportional to rate of change in GPE, water-level measurements can be used in calculating the rate of ground-water flow.

Water-level measurements will be obtained by measuring the depth to water (DTW) in each monitoring well and borehole. DTW measurements will be made from designated points of known elevation on each well or from the ground surface at each borehole. DTW measurements will then be converted to water-level elevations so that water levels across the site can be compared.

Water levels will be accurate to the nearest 0.01 ft (3 mm) for monitoring wells and the nearest 0.1 ft (30 mm) for open boreholes. In order to reduce the effect of temporal fluctuations, all water-level measurements will be taken over as narrow a span of time as practical.



#### 4.3.3 Slug Tests

In most soils, the rate of ground-water flow is a function of the change in GPE and the hydraulic conductivity of the soil. A slug test is a method of determining the hydraulic conductivity of a geologic formation by measuring the rate at which the formation yields water to a well. Slug tests are most suitable for determining average hydraulic conductivities of granular soils.

Slug tests will be performed in all wells installed as part of this investigation. In general, slug tests consist of measuring the fall and rise of water in a well caused by the sudden removal of a slug of water. The specific procedures for performing the slug test are described in the FSP.

#### 4.3.4 Laboratory Permeability Tests

Laboratory permeability tests provide another means of determining hydraulic conductivity in soils. Laboratory permeability tests are more suitable for fine-grained soils or rocks of low permeability. Laboratory permeability tests will be performed in accordance with test method American Society for Testing and Materials (ASTM) D 5084.

Laboratory permeability tests will be performed on approximately ten samples of rock or fine-grained soil. Specific locations and depths of the samples collected will be determined in the field on the basis of conditions encountered. It is anticipated that samples will be collected from buried alluvium, Pennsylvanian shales, fine-grained Pleistocene deposits, and fine-grained fill deposits. Samples will be collected using the Shelby tube method as described in ASTM D 1586.

#### 4.4 River Basin Analysis

Evidence presently available indicates that ground-water flow in the slag deposits is part of the same flow system as the water in the Little Vermilion River. For the purposes of this investigation, the Little Vermilion River system consists of the river water and the associated ground water in the shallow alluvium along the river.

Seeps, tributaries, runoff gullies, and discharge pipes may contribute to flow in the Little Vermilion River system. In order to locate such features, a geologist will traverse the area and make field observations. When such features are identified, they will be located on the map of the area and marked in the field. Identified features will also be described and an estimate of flow recorded.

To the extent practicable, the rate of flow will be measured in the river and in major seeps, tributaries, and discharge pipes. Flow in the river will be measured as closely as possible to the upstream and downstream property boundaries. Flow will be measured as described in the FSP.

#### 4.5 Holding Pond Investigation

The holding pond at the southern end of the Phase II area will be evaluated as a possible source of the elevated levels of zinc and manganese detected in Monitoring Well No. MW2 during GeoSyntec's Preliminary Site Investigation which was completed in October 1992. The holding pond investigation will follow-up the IEPA Site Screening Inspection conducted in November 1991 where metals and PAHs were found in the holding pond sediments.

The holding pond investigation will consist of the following work elements:

- evaluate the extent and thickness of pond sediment;
- evaluate the permeability of the pond sediment; and
- chemically characterize the pond sediment.

Sediment samples will be obtained to determine the thickness and general character of the pond sediments. Approximately three pond sediment samples will be collected for chemical analysis and undisturbed samples of sediment from various depths will be collected for laboratory permeability analysis as described in Section 4.3.4. Samples will be analyzed as described in Section 4.6. The sediment samples will be collected at various locations around the pond. The core samples will extend to the base of the sediment if possible. All recovered samples will be visually examined and logged. The specific sample collection methods are described in the FSP. It is anticipated that sampling will be conducted from a boat.

#### 4.6 Chemical Analysis and Sampling

##### 4.6.1 Introduction

Soil and water samples will be collected for laboratory chemical analysis. For the purpose of this discussion, the term soil includes natural soils, natural rocks, slag, other waste materials in the solid state, or mixtures of any of the above. The purpose of soil sampling will be to determine whether or not the soils contain constituents that might leach into the ground water. The purpose of ground-water sampling is to determine whether or not such constituents are actually present in the ground water.

All samples will be analyzed according to standard USEPA protocols for laboratories in the Contract Laboratory Program (CLP). Full CLP quality assurance packages will be produced for each sample analyzed. The specific laboratory procedures to be used are described in the QAPP.

#### 4.6.2 Analytical Suites

Two different suites of analyses will be used in this investigation: (i) a metals-focused suite; and (ii) a hydrocarbons-focused suite. The metals-focused suite will be performed on all samples collected for chemical analysis. The hydrocarbons-focused suite will be performed on samples collected from the area around Monitoring Well No. G-04, where hydrocarbons have been detected in the soil and ground water. In addition, the sediment samples from the holding pond will be analyzed for the PAH group shown in Table 2c and the metals presented in Table 2a.

The metals-focused suite will include the following: (i) metals that have been shown to be of potential concern at the site (i.e., were detected above Illinois Ground-Water Quality Standards for Class I or Class II Ground Water); (ii) major anions; and (iii) field parameters. The specific components of the metals-focused suite are listed in Table 2.

The hydrocarbons-focused suite will consist of two tiers of analyses. The first tier will be performed on all samples collected for chemical analysis in the vicinity of Monitoring Well No. G-04, and will consist only of total petroleum hydrocarbons (TPH). The TPH analyses will be used for defining the extent of the hydrocarbons and as a basis for designing the monitoring well network (see Section 4.8). Samples collected from the monitoring wells will be subjected to a second tier analysis that is consistent with IEPA Underground Storage Tank (UST) parameters. The specific components of the hydrocarbons-focused suite are listed in Table 2.

#### 4.6.3 Soil/Sediment Samples

Soil samples will be collected from the soil borings and sediment samples will be obtained from the holding pond for chemical analysis in

general accordance with Table 1. This schedule may be modified by the Field Director on the basis of conditions encountered. The procedures to be used for collecting, handling, and managing samples are described in the FSP.

Where soils and sediments are to be analyzed for metals, samples will be collected in duplicate, with one sample being analyzed for total metals, and the other being analyzed using the TCLP. Comparison of data from the two methods gives an indication of the potential mobility of any detected metals.

#### 4.6.4 Water Samples

Water samples for chemical analysis will be collected from the piezometers in general accordance with Table 1. This schedule may be modified by the Field Director on the basis of conditions encountered during the investigation. The procedures to be used for collecting, handling, and managing samples are described in the FSP.

Where water samples are to be analyzed for metals, samples will be collected in duplicate, with one sample being analyzed for total metals and the other being analyzed for dissolved metals. Samples for dissolved metals analysis will be collected by filtering the sample prior to pouring it into the sample bottles. Comparison of data from the two methods gives an indication of whether the metals detected were dissolved in the water or were contained in fine soil particles suspended in the water.

#### 4.6.5 Field Quality Control Samples

Field quality control (QC) samples will be collected in order to assess the potential for sample corruption during collection and handling

and in order to evaluate the repeatability of the findings. Two types of QC samples will be collected and analyzed: (i) equipment blanks; and (ii) duplicates. This QC program is considered the most cost-effective means of preventing false positives resulting from field sampling procedures.

Equipment blanks consist of samples of the water used for decontamination after passing that water through decontaminated tubing, filter equipment, and other devices used in the sample collection process. If analytes are detected in equipment blanks, it indicates the possibility that those analytes may also have been introduced into other samples collected using similar procedures.

Duplicates are a complete second set of samples collected from selected sampling points. Duplicates are assigned separate and arbitrary identification numbers from the original sample so that the laboratory has no indication that the sample is actually a duplicate. One duplicate sample will be collected for every ten samples collected. The determination of which samples will be collected in duplicate will be made by the Field Director. The duplicates will be chosen to represent a variety of the conditions encountered at the site. Equipment blanks will not be duplicated.

#### 4.7 Miscellaneous Investigations

##### 4.7.1 Field Observations

Hydrogeologic field observations will be made throughout the course of the investigation. Particular attention will be given to the following:

- seeps between the former Illinois Central Railroad (ICRR) grade and the Phase I study area;

- seeps along the eastern embankment of the ICRR grade;
- alluvium development along the Little Vermilion River;
- apparent biological diversity and health in the Little Vermilion River;
- the porosity characteristics and compositional variability in the exposed slag deposits; and
- visible hydraulic interconnectivity between the slag deposits and the Little Vermilion River.

#### 4.7.2 Surveying

All soil borings, piezometers, monitoring wells, and sample collection locations will be surveyed by a licensed professional surveyor. Points will be located horizontally to the nearest 1 ft (300 mm). Points on the ground will be located vertically to the nearest 0.1 ft (30 mm). Points on piezometers and monitoring wells, and points used for measuring stream levels, will be located vertically to the nearest 0.01 ft (3 mm). Four points will be vertically located on piezometers and monitoring wells:

- the top of the lid of the protective casing when closed;
- the top of the rim of the protective casing when the lid is open;
- the top of the well casing (TOC) (i.e. the top of the riser); and
- the concrete pad around the well.

## **4.8     Design and Installation of Monitoring Well Network**

### **4.8.1    Introduction**

A ground-water monitoring network for the Phase II area will be designed and installed based upon the findings of the Phase II Site Investigation. This approach will enable the network to be designed, installed, and operated in the most efficient manner possible. It will also ensure that the network provides the most representative and scientifically significant data possible. It is anticipated that this network will consist of approximately five monitoring wells, as discussed in Section 4.8.2. Monitoring wells will be numbered in the G-series, beginning with G-06.

The remainder of this section contains preliminary descriptions of the following: (i) layout and concept of the network; (ii) a general design for the monitoring wells; and (iii) the anticipated ground-water sampling and analysis program for the wells.

### **4.8.2    Network Layout**

The monitoring-well network will be designed for monitoring zones of interest identified during the Phase II Site Investigation. These zones may include the alluvium at the downgradient boundary of the property (approximately two to three wells), any areas along the upgradient property line where constituents may be entering the Phase II area (a minimum of one well), and within or adjacent to identified constituent sources in the Phase II area (possibly one or more wells).



#### 4.8.3 Well Design

It is proposed that the monitoring wells will be constructed of nominal 2-inch diameter, Schedule 40 PVC monitoring well pipe. The intakes for the wells will consist of a 10 ft (3 m) length of no. 10 factory-slotted monitoring well screen. Sections of pipe will be joined using pre-manufactured flush threads.

GeoSyntec believes that PVC, not stainless steel, is the best material for constructing monitoring wells at the Carus Chemical Company site. PVC offers the best combination of chemical resistance and physical durability under the anticipated conditions of the site. Studies reviewed by Nielsen [1991] indicate that modern PVC of potable-water or well-casing grade does not significantly affect ground-water conditions. These studies also indicate a very high degree of resistance to chemical degradation under nearly all types of ground water encountered on hazardous waste sites. The only exceptions noted in these studies were for sites with extremely high concentrations (greater than 100,000  $\mu\text{g/l}$ ) of certain solvents. Such conditions do not exist at the Carus Chemical Company site.

None of the alternative materials for well casing or screen are as suitable for use at the Carus Chemical Company site as PVC. Studies reviewed by Nielsen [1991] indicate stainless steels can leach and adsorb certain metallic ions at the low parts per billion range. Furthermore, stainless steels contain percent-level concentrations of chromium and manganese, which are primary constituents of concern at the Carus Chemical Company site. Flakes, burs, or gouge dust from these metals could become entrained in the water samples and then detected by total metals analyses, thereby giving false positive results. For these reasons, stainless steel is less suitable for use in monitoring wells at the Carus Chemical Company site than is PVC. Fluoropolymer materials are considered unsuitable for use at the Carus Chemical Company site due to mechanical problems inherent with the material: (i) the tendency of

fluoropolymer well screens to deform over time; (ii) the excessive flexibility of the material; and (iii) poor grout-adhesion characteristics.

Annular materials will consist of a gravel pack and grout. The gravel pack will consist of uniformly sized, clean quartz sand of approximately 1 mm size and of a type manufactured for use in well applications. The grout will consist of bentonite chips. Grouts in slurry form will not be used due to anticipated problems with the large void spaces in the slag deposits. A concrete surface seal will be used to divert runoff away from the well. The specific procedures to be used in constructing the wells are described in the FSP.

After the wells have been installed and the grouts given time to set, the wells will be developed using the methods described in the FSP. The purpose of well development is to remove silty material that may be present in the well or filter gravel. Such silt may be a natural part of the water-bearing formation or may be a product of the grinding action of the drilling tools. If large amounts of loose silt are present in the formation, water samples may remain turbid even after development is completed.

#### 4.8.4 Sampling and Analysis

A brief ground-water sampling plan will be prepared following installation of the monitoring well network. At this juncture, preparation of a ground-water sampling plan is not feasible because of the large area and complex hydrogeology of the site. The plan will described the specific procedures to be used for sampling each well and the specific constituents for analysis. It is anticipated that the procedures specified will be very similar to those described in the FSP and that the analytical program will be similar to that described in Section 4.6.

#### 4.9 Management of Investigation-Derived Waste

Investigation-derived waste consists of drill cuttings, drilling fluids, development and purge water from wells, and used decontamination waters. Because of the results of the Phase I Site Investigation, it is assumed that the Phase II Site Investigation will not involve hazardous wastes as defined pursuant to the Resource Conservation and Recovery Act (RCRA). However, the results of the analysis will be reviewed to ascertain if investigation-derived wastes should be considered non-hazardous, if so, they will be disposed of accordingly: waste waters will be managed through the plant waste-water treatment system and drill cuttings will be managed as excavated soil. Any chemicals used in decontamination will either be consumed in use or managed as described in the FSP.

#### 4.10 Data Analysis and Report Preparation

Following completion of the Phase II Site Investigation, a report will be prepared that contains the following: (i) a description of the field and data reduction methods; (ii) a presentation of data; (iii) a discussion of the findings; and (iv) conclusions.

## 5. SCHEDULE

The milestones proposed for implementing the Phase II Site Investigation are presented below.

<u>Work Element</u>	<u>Milestone</u>
Submit Phase II Work Plan to IEPA	28 February 1994
Receive IEPA Comments on Phase II Work Plan	31 March 1994
IEPA Approval of Phase II Work Plan	29 April 1994
Initiate Phase II Investigation	31 May 1994
Complete Phase II Site Investigation	30 July 1994
Submit Phase II Site Investigation Completion Report and Data Validation to IEPA	31 October 1994

The milestone schedule is based on submittal of a first draft work plan for Phase II on 28 February 1994 and IEPA approval of a second draft work plan on 29 April 1994. As may be seen on the milestone schedule, it is anticipated that all studies associated with the Phase II Site Investigation will be completed by 30 July 1994. Based on a 30 July 1994 completion of the Phase II Site Investigation, the summary report analysis and data validation would then be complete by 31 October 1994. This schedule is consistent with the schedule presented to the IEPA in Section 8 of the Phase I Site Investigation Completion Report; however, the submittal date of the Phase II Site Investigation Completion Report has been extended to allow for data validation.

**TABLE 1**  
**DRILLING AND SAMPLING PLAN**  
**PHASE II SITE INVESTIGATION WORK PLAN**  
**CARUS CHEMICAL COMPANY**  
**LASALLE, ILLINOIS**

PIEZOMETER DESIGNATION	TARGET ZONE	BOTTOM-HOLE FORMATION	ESTIMATED DEPTH	SOIL SAMPLES FOR METALS-FOCUSED ANALYSIS	SOIL SAMPLES FOR PERMEABILITY ANALYSIS	ANALYSES FOR WATER SAMPLES	NOTES
P-1	saturated slag and alluvium	top of Pennsylvanian	100 ft	slag @ 30 ft and @60 ft in alluvium	in alluvium	metals-focused	screen in either alluvium or slag, not both
P-2	deepest water-bearing zone above fresh bedrock	top of unweathered Pennsylvanian or Pleistocene fine till	70 ft	slag @ 20 ft and @ 40 ft (slag only)	n/a	metals-focused	
P-3	deepest water-bearing unit above limestone	top of Pennsylvanian limestone or auger refusal	40 ft	n/a	in water-bearing unit, if possible	metals-focused	set offsets in all discrete water-bearing zones
P-4	deepest water-bearing unit above limestone	top of Pennsylvanian limestone or auger refusal	40 ft	n/a	in water-bearing unit, if possible	metals-focused	set offsets in all discrete water-bearing zones
P-5	each water-bearing zone, use offsets if necessary	top of unweathered Pennsylvania	30 ft	n/a	fine till, if present	metals-focused petroleum-focused	set offsets in all discrete water-bearing zones
P-6	each water-bearing zone, use offsets if necessary	top of unweathered Pennsylvanian	30 ft	n/a	fine till, if present	metals-focused petroleum-focused	set offsets in all discrete water-bearing zones
P-7	best water-bearing zone	top of unweathered Pennsylvanian	30 ft	n/a	n/a	metals-focused	
P-8	best water-bearing zone	top of unweathered Pennsylvanian	30 ft	n/a	n/a	metals-focused	

**TABLE 1**  
**DRILLING AND SAMPLING PLAN**  
**PHASE II INVESTIGATION WORK PLAN**  
**CARUS CHEMICAL COMPANY**  
**LASALLE, ILLINOIS (continued)**

PIEZOMETER DESIGNATION	TARGET ZONE	BOTTOM-HOLE FORMATION	ESTIMATED DEPTH	SOIL SAMPLES FOR METALS-FOCUSED ANALYSIS	SOIL SAMPLES FOR PERMEABILITY ANALYSIS	ANALYSES FOR WATER SAMPLES	NOTES
P-9	best water-bearing zone	top of unweathered Pennsylvanian	30 ft	n/a	n/a	metals-focused	
P-10	best water-bearing zone	top of unweathered Pennsylvanian	30 ft	n/a	n/a	metals-focused	
P-11	best water-bearing zone	top of Pennsylvanian limestone	40 ft	n/a	weathered shale	metals-focused	set offsets in all discrete water-bearing zones
P-12	best water-bearing zone	top of Pennsylvanian limestone	40 ft	n/a	weathered shale	metals-focused	set offsets in all discrete water-bearing zones
P-13	good water-bearing zones	top of unweathered Pennsylvanian	50 ft	slag or other industrial waste, if encountered	weathered shale water-bearing fill	metals-focused	
P-14	good water-bearing zones	top of unweathered Pennsylvanian	50 ft	slag or other industrial waste, if encountered	weathered shale water-bearing fill	metals-focused	
P-15	alluvium	unweathered Pennsylvanian	20 ft	alluvium	alluvium fresh shale	metals-focused	
P-16	alluvium	unweathered Pennsylvanian	20 ft	alluvium	alluvium fresh shale	metals-focused	
P-17	slag or alluvium, not both	top of Pennsylvanian	50 ft	alluvium, slag @ 10 ft	weathered Pennsylvanian shale alluvium	metals-focused	set offset if sufficient water in both slag and alluvium
P-18	slag	top of Pennsylvanian	100 ft	alluvium slag @ 30 ft and @ 50 ft	n/a	metals-focused	plug back alluvium

**TABLE 1**  
**DRILLING AND SAMPLING PLAN**  
**PHASE II INVESTIGATION WORK PLAN**  
**CARUS CHEMICAL COMPANY**  
**LASALLE, ILLINOIS (continued)**

PIEZOMETER DESIGNATION	TARGET ZONE	BOTTOM-HOLE FORMATION	ESTIMATED DEPTH	SOIL SAMPLES FOR METALS-FOCUSED ANALYSIS	SOIL SAMPLES FOR PERMEABILITY ANALYSIS	ANALYSES FOR WATER SAMPLES	NOTES
P-19	slag	top of Pennsylvanian	50 ft	slag @ 40 ft	n/a	metals-focused	
P-20	water-bearing zones above limestone	top of Pennsylvanian limestone	40 ft	n/a	n/a	metals-focused petroleum-focused	
P-21	slag	top of Pennsylvanian	50 ft	slag @ 40 ft	n/a	metals-focused	
P-22	slag	top of Pennsylvanian	100 ft	alluvium slag @ 30 ft and @ 50 ft	n/a	metals-focused	plug back alluvium

**SAMPLING POINTS NOT  
ASSOCIATED WITH PIEZOMETERS**

SAMPLING POINT DESIGNATION	NUMBER OF SAMPLES	SAMPLE LOCATION	SAMPLE MATRIX	ANALYTICAL FOCUS
US	2	Little Vermilion River at upstream property line	water	metals
DS	2	Little Vermilion River at downstream property line	water	metals
HP1 through HP3	5	holding pond sediments	sediment	metals and PAH group

n/a denotes not applicable

**TABLE 2**  
**CHEMICAL ANALYSES TO BE PERFORMED**  
**PHASE II SITE INVESTIGATION WORK PLAN**  
**CARUS CHEMICAL COMPANY, LASALLE, ILLINOIS**

**Table 2a: Metals-Focused Suite**

<u>Metals</u>	<u>Anions</u>	<u>Field Parameters</u>
Barium	Sulfate	Conductivity
Cadmium	Sulfide	pH
Chromium	Carbonate	Temperature
Iron	Bicarbonate	Oxidation Potential
Lead	Cyanide	
Manganese		
Mercury		
Nickel		
Zinc		

**Table 2b: Hydrocarbons-Focused Suite: First Tier**

Total Petroleum Hydrocarbons.  
(Metals-Focused Suite will also be performed.)

**Table 2c: Hydrocarbons-Focused Suite: Second Tier**

<u>BTEX Group</u>	<u>PAH Group</u>	<u>UST Metals Group</u>
Benzene	Naphthalene	Arsenic
Toluene	Acenaphthalene	Barium
Ethyl Benzene	Acenaphthene	Cadmium
Xylenes (Total)	Fluorene	Chromium
	Phenanthrene	Lead
	Anthracene	Mercury
	Fluoranthene	Selenium
	Pyrene	
	Benzo(a)anthracene	
	Chrysene	
	Benzo(b)fluoranthene	
	Benzo(k)fluoranthene	
	Benzo(a)pyrene	
	Dibenzo(a,h)anthracene	
	Benzo(g,h,i)perylene	
	Ideno(1,2,3-cd)pyrene	



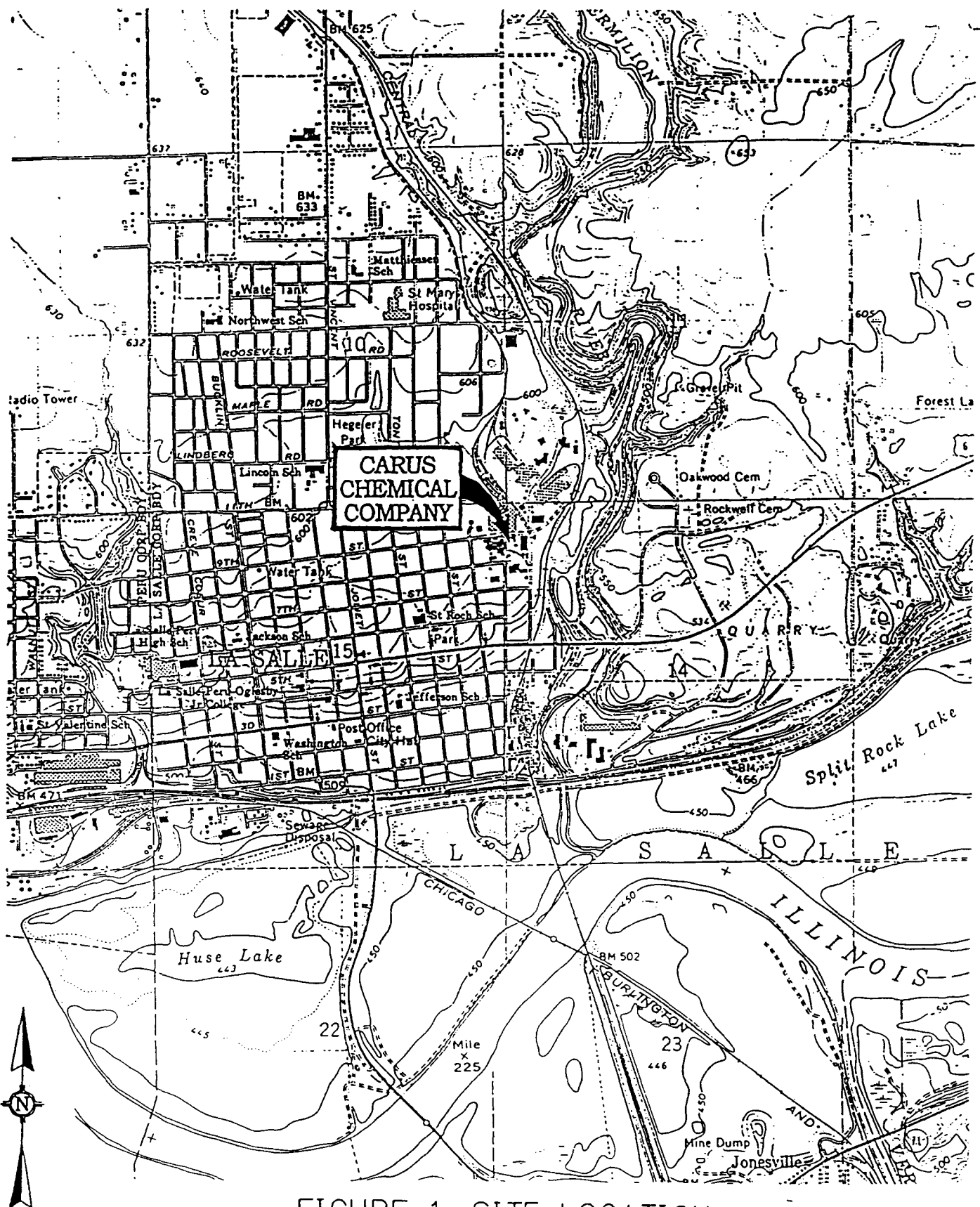
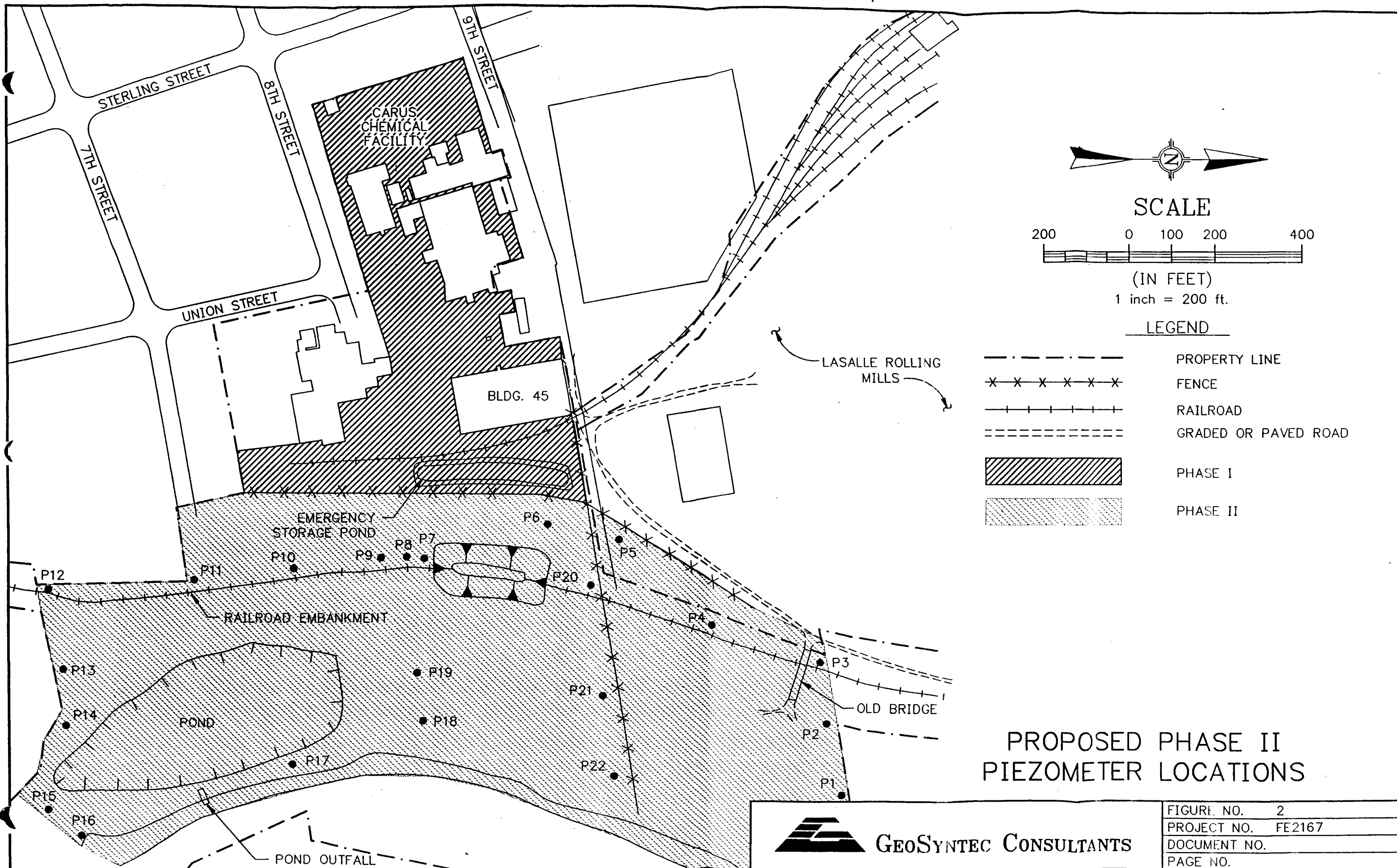


FIGURE 1. SITE LOCATION



GEOSYNTEC CONSULTANTS

FIGURE NO.	1
PROJECT NO.	FE2167
DOCUMENT NO.	
PAGE NO.	



GeoSYNTEC CONSULTANTS

FIGURE NO.	2
PROJECT NO.	FE2167
DOCUMENT NO.	
PAGE NO.	

## APPENDIX A

**APPENDIX A**

**HEALTH AND SAFETY PLAN FOR**

**PHASE II SITE INVESTIGATION**

**CARUS CHEMICAL COMPANY**

**MANUFACTURING FACILITY**

**LASALLE, ILLINOIS**

Prepared for

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Attachment 1 Sign-Off Sheet

## 1. INTRODUCTION

This Health and Safety Plan (HASP) provides guidance for conducting the field activities of the Carus Chemical Company Phase II Site Investigation in a manner that will control the risk to the health and safety of GeoSyntec Consultants' staff, subcontractors and the general public. This document complements the Proposed Work Plan for Phase II Site Investigation (Work Plan), Quality Assurance Project Plan (QAPP), and the Field Sampling Plan (FSP).

The purpose of the HASP is to define the requirements and designate the protocols for protecting personnel and the general public during the Phase II Site Investigation. Applicability extends to all GeoSyntec Consultants employees, subcontractors, and site visitors.

The site investigations are intended, in part, to determine the nature and extent of hazardous substances at the site. Historical information regarding the types of materials that may be encountered at the site has been used to specify medical surveillance program requirements, monitoring/sampling equipment, and personal protective equipment. As the Phase II Site Investigation proceeds, and more detailed information regarding the types, quantities, and extent of hazardous substances becomes known, the HASP may be modified accordingly.

This plan will be reviewed and an acknowledgement signed by all personnel prior to entering an exclusion zone or contamination reduction zone, requiring them to comply with plans and procedures contained herein. A sign-off sheet is provided as Attachment I to this document.

The work on site will be performed in accordance with the GeoSyntec Consultants Corporate Health and Safety Program and policies, and all applicable and appropriate federal, state, and local policies and regulations.



## **2. GENERAL PROCEDURES**

The following general health and safety procedures apply to all activities conducted at the Carus Chemical Company manufacturing facility. They provide the "umbrella" under which the task-specific health and safety plan was developed. The plan has been developed in compliance with 29 CFR 1910.120, with guidance from the NIOSH/OSHA/USCG/EPA Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities and the EPA Standard Operating Safety Guides.

### **2.1 Responsibilities/Authorities**

This section describes the responsibilities and authorities of project personnel with regard to health and safety. Ultimately, the health and safety for project personnel lies with the individual. All personnel need to be cognizant of the hazards and the methods of reducing the risk of injury and illness. All personnel will comply with the rules and procedures set forth in this plan and will make project management aware of any conditions which may jeopardize the welfare of project workers and/or the general public.

#### **2.1.1 Project Principal - Neil D. Williams, Ph.D., P.E.**

The Project Principal is responsible for management oversight of the project.

#### **2.1.2 Project Manager - Nandra D. Weeks, P.E.**

The Project Manager is responsible for the management of all aspects of the project, including health and safety, ensuring that all project tasks receive appropriate health and safety review before commencement

of field activities, and ensuring that the necessary equipment and facilities are available to implement the health and safety plan.

#### **2.1.3 Field Director**

The Field Director is responsible for ensuring that the health and safety aspects for his particular tasks are addressed, for ensuring that site subcontractors follow the HASP, for implementation of the appropriate work practices and for notifying the Health and Safety Coordinator of any changes in work conditions which affect the health and safety aspects of the task.

#### **2.1.4 Health and Safety Coordinator - Ms. Nandra D. Weeks, P.E.**

The Project Manager is also assigned as the Health and Safety Coordinator for the project. The Health and Safety Coordinator is responsible for preparation of the HASP and the task-specific health and safety plan. She is also responsible for making modifications to the plan and recommending changes to the field tasks if there are health and safety ramifications, and for ensuring that all required health and safety sampling/monitoring is performed and the required health and safety documentation is maintained. The Health and Safety Coordinator will assign site specific tasks to the Field Director for implementation.

#### **2.1.5 Site Health and Safety Officer (SHSO) - Mr. Jack Raymer**

The SHSO is responsible for the on-site implementation of the HASP, including the task-specific health and safety plan. The SHSO or his designate has the authority to modify or stop any work on the site if it represents an imminent danger to the health and safety of site workers or the general public. Due to the size of this project, the Field Director will assume the duties as SHSO.

#### 2.1.6 Subcontractors

All subcontractors are required to abide by the specifications of this and the task-specific health and safety plan. They also are required to comply with all applicable and appropriate federal, state, and local laws, standards, and regulations.

### 2.2 Hazard Analysis and Control

The information in this section is based upon data from earlier investigations and published information.

#### 2.2.1 Chemical Hazards

Earlier investigations regarding the Carus Chemical Company site indicate that ground water in several areas is minimally impacted by inorganics which include trace metals such as cadmium, lead, and mercury.

Organic compounds have also been found in relatively low concentrations primarily in the pond sediment. In addition, due to historical accounts of an underground storage tank (UST) hazardous substances such as benzene, toluene, ethylbenzene and xylene (BTEX) may be encountered in the northeastern corner of the Phase I Site Investigation area.

Activities during the site investigations are not expected to disturb large areas of contaminated materials; therefore, exposures to hazardous substances are expected to be limited to close proximity of the work activity.

Exposure criteria for known contaminants on-site are expressed below as the Permissible Exposure Limits (PELs) established by OSHA and detailed under 29 CFR 1910.1000:

<u>Contaminant</u>	<u>PEL</u>
Benzene	1.0 ppm*
Methylene Chloride	50 ppm
Ethylbenzene	100 ppm
Tolulene	100 ppm
Xylene	100 ppm
Mercury	0.1 mg/m <sup>3</sup> **

\* STEL is 5 ppm. This is the permissible level for a 15 minute exposure. The action level is 0.5 ppm.

\*\* Ceiling (not-to-exceed) concentrations.

### 2.2.2 Physical Hazards

There are various physical hazards that project personnel may be exposed to during the field investigations. These include holes or ditches, sharp objects, slippery surfaces, uneven terrain, tools and equipment, and machinery. Other physical hazards include automobile traffic on nearby roads that are near the site. Specific arrangements will be made when work must be performed within or near the right-of-ways of the roads. These include notification of the appropriate parties and use of signs and high-visibility vests (orange).

Weather conditions may expose personnel to extremes of heat or cold. The principal hazards of cold stress are frostbite, hypothermia, and impaired ability to work. Wind will lower the effective temperature.

The use of personal protective clothing will increase the risk of heat stress. Heat stress can result in unacclimated persons, in

particular, and can cause rashes, cramps, discomfort, and dehydration. Heat stroke is the most dangerous form of heat illness and requires immediate medical attention. The SHSO must observe for conditions which promote heat or cold stress illnesses.

The use of power tools and equipment, particularly drilling equipment, often creates excessive noise. Chronic overexposure at levels greater than 85 decibels can lead to loss of hearing. At the least, excessive noise can annoy or distract workers and increase the risk of other accidents due to interference with communication.

Other physical hazards include mechanical and moving equipment (especially augers), and flammable atmospheres.

### 2.2.3 Hazard Control

Engineering controls are preferred to control hazards whenever such controls are available and practical. The use of dust suppression techniques, equipment guards, and work procedures that minimize worker exposure to hazardous substances or situations are examples of engineering controls.

Only equipment that is used for its intended task and that is in safe operating condition will be used. Personnel will be familiar with the hazards associated with the use of the tools and equipment and methods to mitigate the hazards.

Personal protective equipment (PPE) will be utilized when engineering and administrative controls are not feasible or practical. Personal protective equipment may consist of boots, clothing, gloves, and head and hearing protection. Respirators may be utilized if concentrations of airborne contaminants warrant. All respirators will be NIOSH/MSHA approved, and all workers will have been properly trained and fit-tested.

Adequate cold weather clothing and work breaks will be utilized to prevent cold stress. Heat stress will be controlled through the use of monitoring, modification of work schedules, and preventive techniques, as appropriate.

## **2.3        Training**

### **2.3.1        Basic OSHA Training**

All personnel will have received the health and safety training as described in this section before being allowed to participate in field activities that could expose them to hazardous substances, safety hazards, or health hazards. This training is required pursuant to 29 CFR Part 1910.120(e).

- *Forty-Hour Hazardous Waste Operations Health and Safety Training*- Forty hours of classroom instruction and simulated field exercises regarding the following topics: (i) biology, chemistry, and physics of hazardous materials; (ii) toxicology; (iii) industrial hygiene; (iv) hazard evaluation and control; (v) PPE; (vi) medical surveillance; (vii) decontamination; (viii) legal and regulatory aspects; and (ix) emergency response.
- *Eight-Hour Annual Waste Operations Health and Safety Refresher Training* - Eight hours of refresher training annually, as necessary.

### **2.3.2        Site- and Task-Specific Training**

#### *Site-Specific Health and Safety Training*

Health and safety instruction regarding the following Carus Chemical Company site-specific topics will be given to all project personnel: (i)

names of personnel and alternates responsible for site safety and health; (ii) safety, health, and other hazards present on the site; (iii) PPE requirements; (iv) standard operating procedures; (v) hazard control; (vi) medical surveillance; (vii) site- and task-specific health and safety plans; (viii) site emergency procedures; and (ix) routine activities.

#### *Task-Specific Health and Safety*

Site personnel will receive training regarding health and safety aspects of the work prior to the commencement of any new tasks. This information will be conveyed during weekly safety meetings, or more often, as required.

#### **2.4      Medical Surveillance**

All project personnel who may engage in on-site activities for 30 days or more per year will participate in a medical surveillance program as described in this section. The medical surveillance program consists of a baseline or initial medical examination, an annual medical examination, a termination examination, and episodic medical examinations, as necessary.

All exams will be performed by a licensed physician. Initial exams shall include:

- history and physical;
- executive profile;
- full vision screen;
- urinalysis;
- EKG;
- spirometry;
- chest X-ray (2 views);
- audiometry;

- hemocult slides;
- T.B. skin test and tetanus toxoid; and
- flexible sigmoid (if needed).

Annual exams include:

- history and physical;
- full vision screen;
- urinalysis;
- executive profile;
- hemocult slides;
- EKG (if indicated);
- chest X-ray (two views, if indicated);
- audiometry (if indicated); and
- flexible sigmoid (if indicated).

An episodic examination will be required if any worker develops signs or symptoms related to the possible overexposure to hazardous substances or other health hazards, or if an unprotected worker is exposed in an emergency situation. Hence, should any worker believe that exposure has occurred at site, the Project Manager must be advised immediately. The scope of any episodic examinations will be left to the discretion of the examining physician.

Medical records for employees who participate in the medical surveillance program will be kept on file for 30 years after tenure or as required by OSHA. All employees have access to their medical records.

## 2.5 Work Zones and Monitoring

### 2.5.1 Work Zones

Where there is a potential for the accidental spread of hazardous substances to clean areas, work zones will be established where different



types of operations will occur in each work zone, and the flow of personnel and equipment will be controlled. The establishment of work zones will help ensure that personnel are properly protected against hazards present where they are working, that work activities and contamination are confined to the appropriate areas, and that personnel can be located and evacuated in an emergency.

Prior to the commencement of field activities, work zones will be established by means of tape, barriers, or other methods to clearly delineate them and to meet operational and safety objectives.

#### *Exclusion (Controlled) Zone*

The exclusion zone is the area where hazardous substances or safety hazards are present or are expected to occur. Entry into this area is limited to those personnel wearing the specified PPE, who have completed the required health and safety training, and who are participating in the medical surveillance program (if necessary). The boundary of the exclusion zone will be determined for each individual task and may change depending on site activities and conditions. The exclusion zone will be clearly delineated through the use of signs, barricade tape, and/or other means. Access control points will be established to regulate the flow of personnel and equipment into and out of the zone and to help verify that proper procedures for entering and exiting are followed. The required level of PPE in the exclusion zone depends upon the job assignment, and will be specified in the task-specific health and safety plan.

#### *Contamination Reduction Zone*

The contamination reduction zone is the transition area between the contaminated area and the clean area. This zone is designed to reduce the probability that the clean support zone will become contaminated or affected by other site hazards. Decontamination of personnel and equipment will occur in the contamination reduction zone. Personnel and

equipment will not be allowed to leave the contamination reduction and exclusion zones without being properly decontaminated, except in emergency situations.

### *Support Zone*

The support zone is all areas outside the exclusion and contamination reduction zones. The support zone includes the Field Operations Center where logs will be maintained recording the names of personnel and level of protection for all entries into the exclusion and contamination reduction zones. A decontamination facility will be established in the support zone.

### **2.5.2 Monitoring**

Monitoring will be performed to assess the exposure to hazardous substances and to ensure that the proper PPE has been selected. It will also be performed to delineate areas where protection is needed.

The task-specific health and safety plan describes specific monitoring/sampling appropriate for the work elements of the Phase II Site Investigation.

Air monitoring/sampling may be performed using two approaches, as necessary; the use of direct-reading real-time instruments and the collection of air samples in a suitable collection medium and subsequent laboratory analysis. Alternatively, air samples may be collected and analyzed according to the NIOSH Manual of Analytical Methods, Third Edition (as applicable). It is anticipated that the following air contaminants and conditions may occur at some locations of the site: organic vapors and dusts. In consideration of these potential air contaminants and conditions it is planned that direct reading instruments will be used in areas of concern. Table 1 describes the appropriate response action for varying organic vapor conditions.

## 2.6 Personal Protective Equipment (PPE)

This section describes the personal protective equipment program for the entire project. The level of PPE required will be dependent upon the work element and site hazards and may vary from task to task. Modifications (i.e., upgrading/downgrading) of the specified levels of PPE may be made at the discretion of the Site Health and Safety Officer or his designate as more information regarding site hazards becomes known. As previously discussed, engineering controls and work practices will be the primary methods of protecting site workers. Only when such controls and practices are not feasible will PPE be utilized. Hearing protection will be provided for those tasks where the noise levels warrant the use of hearing protection, regardless of the level of protection utilized.

### 2.6.1 Level B

Level B will be selected when the highest level of respiratory protection is necessary.

#### Level B

- Pressure-demand, full-face piece SCBA or pressure-demand supplied-air respirator with escape SCBA;
- Chemical-resistant coveralls (polyethylene-coated Tyvek or equivalent);
- Gloves, outer, chemical-resistant (nitrile or equivalent);
- Gloves, inner, chemical-resistant (latex or equivalent);
- Boots, chemical-resistant, steel toe and shank (PVC/polyurethane or equivalent);
- Hard hat; and
- Cotton coveralls\*.

\* Optional

### 2.6.2 Level C

Level C or Level C - modified will be selected when the concentration and type of air contaminant is known and the criteria for selection of air-purifying respirators are met. These criteria are listed on Table 1, Air Monitoring Action Levels, Organic Vapor Readings. Cartridges will be replaced after 8 hours of use.

#### Level C

- Full-face air-purifying respirator with appropriate cartridges/canisters (combination dust/fume/mist and organic vapor or equivalent). The exact model/stock number of this equipment will be determined by the project Site Health and Safety Coordinator.
- Chemical-resistant coveralls and booties (polyethylene-coated Tyvek or equivalent). All joints must be taped.
- Gloves, outer, chemical-resistant (nitrile or equivalent).
- Gloves, inner, chemical-resistant (latex or equivalent).
- Boots, steel toe and shank (PVC polyurethane or equivalent equipment).
- Hard hat.
- Cotton coveralls\*.

\*optional

#### Level C - Modified

- Disposable coveralls and booties (Tyvek or equivalent). All joints must be taped.

- Gloves, chemical-resistant (latex or equivalent).
- Boots, steel toe.
- Hard hat.
- Safety glasses with side shields.
- Half-face air purifying respirator with appropriate cartridges. Full-face respirators available.
- Cotton overalls\*.

\*optional

#### 2.6.3 Level D

Level D to be selected when minimal protection is required from air or contact contaminants.

##### Level D

- Disposable coveralls (Tyvek or equivalent);
- Gloves (latex or equivalent);
- Boots, steel toe;
- Hard hat; and
- Safety glasses with side shields.

#### **Level D - Modified**

- Coveralls, cotton;
- Gloves, cotton;
- Boots, steel toe;
- Hard hat; and
- Safety glasses with side shields.

#### **2.6.4 Maintenance, Storage, and Inspection of PPE**

All PPE used at the site will be stored and maintained in the site office. Disposable items will be inventoried on a regular basis in order to ensure that an adequate supply is always available. The Health and Safety Officer or the designated representative will routinely inspect all PPE and monitoring equipment to ensure that it is in good operating condition.

#### **2.6.5 Training and Use of PPE**

All personnel working at the site will have completed the OSHA-required 40-hour Safety and Hazardous Waste Sites course described in Section 2.3.1. This course includes training in the use of PPE and monitoring equipment. The course also includes fit-testing of respirators. Equipment for fit-testing will be kept on-site in the event that it is necessary to re-test any personnel.

All personnel will don the required PPE before entering the exclusion zone. The Health and Safety Officer or his designate will be responsible for ensuring all required PPE is worn. Personnel working in the exclusion zone will be responsible for monitoring each other to ensure that the PPE remains in good condition during the work period (e.g., Tyvek is not torn, etc). Personnel will remove PPE in the contamination-reduction zone as described in Section 2.5.1, Work Zones. Washing of boots and removal of PPE will occur in the contamination reduction zone.

The Health and Safety Officer or his designate will be responsible for evaluating the PPE program to ensure that all necessary equipment is on-site, in good working condition, and is being used properly.

#### **2.6.6 Work Mission Duration**

During activities that require Level C PPE, all personnel will, at a minimum, take breaks according to the following schedule:

- 15 minute break mid-morning;
- 30 minute break at lunch; and
- 15 minute break mid-afternoon.

Because the risk of heat stress is increased when working in Level C, more frequent breaks will be taken during warm weather conditions. All personnel will be encourage to take breaks whenever the symptoms of heat stress (dizziness, headaches, excessive sweating, weakness) occur. All personnel will monitor each other for the symptoms of heat stress.

## 2.7 Equipment Decontamination

All equipment/tools used in the exclusion zone will be inspected for contamination prior to removal from the site. Any equipment/tools with visible contamination will be cleaned. A water and detergent solution will be used for highly contaminated equipment, followed by a high-pressure hot water rinse. All water used during decontamination will be contained for testing and proper disposal. If necessary, cleaning solvents (approved by the Site Health and Safety Coordinator) may be used on a case-by-case basis. Personal protective clothing will be double-bagged in plastic bags and disposed of as described in the FSP.

## 2.8 Emergency Preparedness

### 2.8.1 Overview

This section describes the contingency plans to be implemented in the event of injuries, illness, accidents, fires and/or releases of hazardous substances. The contingency plan provides for guidelines for the proper response to emergency situations, but the actual response will depend on the situation.

The following equipment will be available on site for emergencies;

- First-aid kit;
- 20 lb ABC-rated fire extinguisher;
- Personal protective equipment, including air purifying respirators and appropriate cartridges; and
- Eyewash and charged water hose.



An emergency response protocol will be established for the site and coordinated by the Site Health and Safety Officer. Personnel will be informed of emergency response protocols and phone numbers and/or locations where assistance can be obtained. Table 2 lists the contacts which will be established prior to the start of the project.

#### **2.8.2 Emergency Decontamination**

In the event of personal injury, the primary objective is to remove the individual from the area and administer first aid with follow-up medical treatment at a clinic or hospital. If the injury/illness occurs within the exclusion zone, then decontamination will be performed. Figure 1 presents the emergency decontamination logic which will be followed in the event such decontamination must be considered.

#### **2.8.3 First Aid**

There will be a minimum of one worker with current certification in first aid/CPR training at the site at all times. There will be at least one first-aid kit of adequate size available for each concurrent task. Arrangements with local emergency response organizations will be made prior to the commencement of field activities and all field personnel will be made aware of methods/numbers to contact these groups.

#### **2.8.4 Fires**

If possible, fires will be extinguished utilizing on-site fire extinguishers. Site workers are not trained fire fighters and should not attempt to extinguish fires beyond reasonable attempts given the combustible nature of most protective clothing. The local fire

department will be called for assistance if a fire is obviously out of control or cannot be put out using on-site equipment. Notification will include any other hazards the fire department should be aware of when they respond.

#### 2.8.5 Notification/Reporting Requirements

All injuries/illnesses, regardless of severity, will be reported to the Project Manager, the GeoSyntec Consultants Health and Safety Coordinator, and the Carus Chemical Company Project Coordinator as soon as practical following the incident. If the incident results in a lost time accident, appropriate notification will be made using worker's compensation First Report of Injury forms. All accidents that result in property damage will be reported as soon as practical following the incident. All injuries/illnesses will be investigated and notification/reporting requirements met per standard GeoSyntec Consultants and Carus Chemical Company policy and procedure.

#### 2.8.6 Location and Route to Local Hospital

Directions to the hospital from Carus are as follows:

- go West from the Carus Chemical Company on 8th Street 5 blocks to Joliet St.;
- go South on Joliet 5 blocks to 3rd St (U.S. 6);
- go West on 3rd St (U.S. 6) 19 blocks through LaSalle and Peru to West St;
- go North on West St. 2 blocks to hospital; and
- turn into the emergency room entrance.

### 2.8.7 Spill Containment

Containerized or drummed hazardous or potentially hazardous liquids and/or solids will be stored on plastic to prevent the contact of these materials with soils, ground water, or surface water in the event of a spill or leakage. The containers will be periodically inspected in order to ensure that leakage is not occurring. Similarly, when these materials are transferred, plastic will be placed over the ground around the transfer zone. A supply of commercially-procured absorbent material will be present if liquids are being transferred and will be maintained at the FOHQ throughout the project.

Spill containment during any transport and disposal of investigation-derived waste materials will be the responsibility of the waste transporters and disposal facility. Only licensed hazardous waste transporters and appropriately permitted disposal facilities will be used for disposal of the materials.

### 2.9 Safe Work Practices

This section describes work practices that are designed to ensure the safety and well-being of personnel engaged in field activities:

- All work will be performed in accordance with GeoSyntec Consultants standard policies and procedures, federal, state, and local rules and regulations with regards to health and safety.
- There will be no eating, drinking, smoking, and chewing gum or tobacco within an exclusion (control) zone or a contamination reduction zone.

- All work will be performed pursuant to the applicable OSHA regulations, 29 CFR 1910 and 1926.
- If unknown wastes (drums, barrels, etc.) are discovered on site, they will not be disturbed until the appropriate personal protection requirements are identified for the task.
- No intrusive activities will commence without first determining whether underground utilities (water, sewer, power, etc.) may be encountered. This determination may be accomplished through the use of utility maps or hand probes to definitively establish the presence and location of these hazards.
- Two-way radios will be used for site communications, if needed. Additionally, the GeoSyntec Consultants field operations headquarters (FOHQ) will be equipped with a telephone.

### 3. TASK-SPECIFIC HEALTH AND SAFETY PLAN

#### 3.1 Task Descriptions

The work elements associated with the Phase II Site Investigation are discussed in Section 4 of the Work Plan. The primary work elements are as follows:

- drilling and piezometer installation;
- hydrogeologic testing;
- river basin analysis;
- holding pond investigation;
- chemical analysis and sampling;
- field observations;
- surveying; and
- design and installation of monitoring well network.

#### 3.2 Task Hazard Assessments

Task activities will require personnel to come into direct contact with soil and ground water potentially contaminated with hazardous substances. These substances, as identified in earlier investigations, include volatile organic compounds. Disturbance of ground water and soil contaminated with volatiles represent an inhalation hazard. Accumulation of volatile organic compounds in the drill hole may result in a

flammability hazard. Physical hazards include tripping and falling, "pinch" points, falling objects, rotating augers and gears of the drill rig, and heat and cold stress. The drill rig must be inspected each day especially making note of the condition of ropes, emergency cut-off switches, hydraulic leaks, tire inflation, muffler condition, and power transmission linkages. The SSHO will approve the use of this equipment.

In keeping with good industrial hygiene practice, personnel will position themselves upwind or crosswind of augers emerging from holes and during drilling as this is the most likely time for a vapor release. Also, personnel in close proximity to the drilling operations should remain there only if they are actually required for the operation and have a need to be present.

### 3.3 Protective Measures/Equipment

#### 3.3.1 PPE

The following PPE will be utilized during the Phase II Site Investigation.

##### *Level D - Modified*

- Disposable coveralls (Tyvek or equivalent);
- Hard hat;
- Gloves (latex); and
- Steel-toed boots.

Hearing protection may be required of some personnel during this task depending on the condition of the drill rig.

### 3.3.2 Work Zones

An exclusion zone will be established to include 25 ft (8 m) in all directions around the drill rig/well. A contamination reduction zone will be established at the boundary of the exclusion zone either upwind or crosswind. The appropriate PPE for the task will be required for entry into the exclusion zone.

### 3.3.3 Air Monitoring

Given the low levels of organic contaminants known to be at the site, respiratory protection is not expected to be required. However, air monitoring will be undertaken in suspect areas to assess any fugitive organic and other vapor emissions. No environmental air samples will be collected for laboratory analyses. If air monitoring indicates organic vapor readings greater than 1 ppm, PPE levels will be upgraded in accordance with Table 1.

#### 3.3.3.1 Monitoring Program

A photo-ionization detector (PID) or flame-ionization detector (FID) will be used to assess worker exposure to organic vapors in suspect areas and to determine the appropriate level of respiratory protection. The PID, if used, will be equipped with a lamp which is rated at an energy level capable of detecting chlorinated hydrocarbons. Breathing zone measurements will be made at least every 10 min. during drilling. If monitoring indicates the organic vapor concentrations exceed the levels

specified in Table 1 of the Site Health and Safety Plan, then appropriate upgrading of the level of PPE utilized will be implemented.

All parameters measured in the exclusion zone will also be measured in the ambient air (in the support zone or contamination reduction zone) at least every 30 min., and before entering the exclusion zone for the first time each day.

Successful suppression of dust will eliminate the need for monitoring of the trace metals at the site. Should unavoidable dusting be encountered, monitoring will be initiated for characterization.

#### 3.3.3.2 Instrument Calibration and Maintenance

All air monitoring equipment will be calibrated daily according to the manufactures instructions. Operation manuals for all equipment will be kept in the field office trailer. Calibrations and instrument settings will be recorded in the site Health and Safety Log.

The calibration procedures for the Organic Vapor Analyzer (OVA) Model 128 and the HNu Photo Ionization Analyzer Model PI 101 are outlined below.

All instruments will be maintained as specified by the manufacturer.

##### *Century OVA Model 128:*

1. Place instrument in normal operation mode.  
Set CALIBRATE switch to 10x and GAS SELECT to 300.
2. Use CALIBRATE ADJUST (zero) knob to adjust the meter reading to zero.



3. Introduce a methane sample of a known concentration (between 90 and 100 ppm not to exceed 100 ppm) and, if necessary, adjust trimpot R-32 so the meter reading corresponds to the known sample.
4. If the calibration was adjusted in Step 3: Turn off Hydrogen supply valve to put out flame.
5. Leave CALIBRATE switch on X10 position and use calibrate adjust knob to adjust meter reading to 4 ppm.
6. Place CALIBRATE switch to X1 position and use trimpot R-31 to adjust meter reading to 4 ppm.
7. Move CALIBRATE switch to X10 position again. Use CALIBRATE adjust knob to adjust to 40 ppm.
8. Move CALIBRATE switch to X100 position and use trimpot R-33 to adjust meter reading to 40 ppm.
9. Move CALIBRATE adjust knob to adjust meter reading to zero.

*HNu PI 101:*

1. Check battery - Turn function switch to BATT. Needle should go to green region.
2. Zero set - Turn function switch to STANDBY. Set Zero point with the ZERO set control.
3. Turn the function switch to 10X.

4. Introduce the calibration gas (isobutylene) to the instrument and adjust the SPAN control setting as required to read the ppm concentration of the standard.

Table 1

### Air Monitoring Action Levels Organic Vapor Readings

Breathing Zone Level\*Required Action

Less than 1 ppm\*\*

- Level D or Modified Level D protection
- Respirators optional but must be available

1-25 ppm

- Determine possible interference (e.g., vehicle exhaust)
- Level C protection
- Monitor area for determining respirator use boundaries and mark limits
- Evacuate downwind personnel without respirators
- Notify Site Health and Safety Officer
- Continue routine monitoring

Greater than 25 ppm

- Evaluate for interference
- Level B (Supplied-air respirators), required in control zone
- Notify Site Health and Safety Officer and Health and Safety Coordinator
- Evacuate personnel for whom respiratory protection is unavailable
- Determine supplied-air boundaries and perimeter monitoring
- Continue monitoring

Greater than 50 ppm

- Cease disturbing contaminated material
- Notify Site Health and Safety Officer and Health and Safety Coordinator
- Establish levels at downwind property boundary

\* For ten consecutive minutes, at personnel breathing zones, measured with a calibrated photo-ionization detector or flame-ionization detector. These levels are to be above background levels.

\*\* This level is based on the 1 ppm PEL for benzene. Should definitive air monitoring conclude that benzene is not present, this level will be adjusted accordingly. This adjustment will consider other identified contaminants.

It is important to note that transient peaks may exceed action levels, especially when measured at ground level. The Site Health and Safety Officer will be consulted for appropriate personnel response.

**Table 2**  
**Emergency Telephone Numbers**

Ambulance	911
Fire	223-2121
IVCH Hospital	223-3300
Police	223-2131
Sheriff	433-2161
Highway Patrol	224-1150
Illinois Valley Eye Clinic	224-2777
Ill Emergency Services & Disaster Agy	1-800-782-7860
National Response Center	1-800-424-8802
Chemtrec	1-800-424-9300
Health & Safety Coordinator Nandra Weeks, GeoSyntec Consultants	(407) 995-0900

Directions to the hospital from Carus are as follows:

Go West from Carus Chemical Company on 8th Street 5 blocks to Joliet Street.  
Go South on Joliet 5 blocks to 3rd St (U.S. 6).  
Go West on 3rd Street (U.S. 6) 19 blocks through LaSalle and Peru to West Street.  
Go North on West Street 2 blocks to hospital.  
Turn into the emergency room entrance.

**ATTACHMENT 1**

**SIGN-OFF SHEET**

## ATTACHMENT 1

Acknowledgement Signatures

I have read and reviewed the Health and Safety Plan for the Site Investigation at Carus Chemical Company manufacturing facility and Understand the information presented. By signing below, I hereby agree to comply with the provisions contained therein.

_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date
_____ Name	_____ Organization	_____ Date

## APPENDIX B

**APPENDIX B**

**QUALITY ASSURANCE PROJECT PLAN FOR**

**PHASE II SITE INVESTIGATION**

**CARUS CHEMICAL COMPANY MANUFACTURING FACILITY**

**LASALLE, ILLINOIS**

Submitted to

State of Illinois  
Illinois Environmental Protection Agency  
2200 Churchill Road  
Springfield, Illinois 62794-9276

Prepared by

GeoSyntec Consultants  
621 N.W. 53rd Street  
Suite 650  
Boca Raton, Florida 33487

February 1994



**PHASE II SITE INVESTIGATION**  
**CARUS CHEMICAL COMPANY MANUFACTURING FACILITY**  
**LASALLE, ILLINOIS**  
**QUALITY ASSURANCE PROJECT PLAN**

Date: \_\_\_\_\_  
Revision: \_\_\_\_\_

Approval for Implementation

Name: Ms. Nandra D. Weeks, P.E.  
Title: GeoSyntec Consultants Project Quality Assurance Manager  
Signature: \_\_\_\_\_

Approval for Implementation

Name: Dr. Neil D. Williams, P.E.  
Title: GeoSyntec Consultants Project Principal  
Signature: \_\_\_\_\_

Approval for Winston & Strawn

Name: Mr. Mark Sargis, Esq.  
Title: Project Counsel  
Signature: \_\_\_\_\_

Approval for Carus Chemical Company

Name: Mr. Roger C. Threde  
Title: Carus Chemical Company Project Coordinator  
Signature: \_\_\_\_\_

Approval for Implementation

Name: \_\_\_\_\_  
Title: IEPA Project Official  
Signature: \_\_\_\_\_

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Table 1. Primary Subcontractor Support

Figure B-1: Project Organization Chart

ATTACHMENT 1: LABORATORY QUALITY ASSURANCE PROJECT PLAN

ATTACHMENT 2: FIELD SAMPLING PLAN

### 3. PROJECT DESCRIPTION

The Quality Assurance Project Plan (QAPP) for the Phase II Site Investigation at the Carus Chemical Company manufacturing facility has been developed to provide management control that will ensure that all technical data generated during the Phase II Site Investigation is accurate, defensible, and representative of conditions encountered at the site. GeoSyntec Consultants of Boca Raton, Florida, is manager of the Phase II Site Investigation. Specialty subcontracting services are being provided by ARDL, Inc. (analytical laboratory), and Layne Environmental (drilling).

The purposes of the Phase II Site Investigation are presented in Section 1.2 of the Work Plan. The Phase II Site Investigation area includes Carus Chemical Company property located east of the main plant area. The scope of work associated with the Phase II Site Investigation includes:

- environmental field work, such as drilling, sampling, and hydrogeologic testing;
- laboratory analysis of ground water, surface water, soil, and sediment for constituents of concern;
- design, installation, and testing of a monitoring-well network; and
- data analysis and report preparation.

#### 4. PROJECT MANAGEMENT AND ORGANIZATION

Winston & Strawn has retained GeoSyntec Consultants to prepare the required documents for the Phase II Site Investigation at the Carus Chemical Company manufacturing facility and to conduct that work.

The Carus Chemical Company Project Coordinator, Mr. Roger C. Threde, will serve as primary liaison with Illinois Environmental Protection Agency (IEPA) and will receive direct reports from the GeoSyntec Consultants' Project Principal, Dr. Neil D. Williams, P.E. GeoSyntec Consultants will be responsible for the execution of all work associated with implementation of the Phase II Site Investigation, to include:

- preparation of the Work Plan and related project documents;
- hiring of subcontractors for drilling, analytical services, data validation, and other specialty services as may be identified throughout the course of the project; and
- completion of the Phase II Site Investigation in accordance with the Work Plan.

##### 4.1 General Project Organization

To provide the necessary attention and control to the various tasks in the Work Plan, GeoSyntec Consultants has assigned a Field Engineer and Field Geologist to be accountable to the Project Manager. The Field Engineer and Field Geologist will have responsibility for Work Plan task completion.

The project organizational structure and personnel responsibilities are organized to assure that quality assurance procedures are followed by those who have been assigned to perform the work and that conformance

with these procedures is verified by quality assurance personnel who are not directly responsible for performing the work.

- Project Manager: The GeoSyntec Consultants Project Manager reports directly to the Project Principal of GeoSyntec Consultants. This person has been assigned authority and responsibility to coordinate and implement technical procedures and work activities prescribed by the project scope of work. The Project Manager is responsible for ensuring that project activities are conducted in compliance with the Work Plan as well as federal, state, and local codes and regulations. The Project Manager ensures that quality related documents contain the necessary technical information and instructions consistent with the applicable requirement of the contract and the QAPP.
- Health and Safety Coordinator: The GeoSyntec Consultants' Project Manager will also perform as Health and Safety Coordinator for the Phase II Site Investigation. In this capacity, the Health and Safety Coordinator is responsible for preparation of the site Health and Safety Plan and general oversight of the health and safety related aspects of the work. The Health and Safety Coordinator is also responsible for making modifications to the plans and recommending changes to the field tasks if there are health and safety ramifications. The Health and Safety Coordinator is responsible for ensuring that all required health and safety sampling/monitoring is performed and that the required documentation is maintained. The Health and Safety Coordinator may delegate some tasks to the Site Health and Safety Officer for implementation.
- Field Engineers and/or Geologists: The Field Engineer and Field Geologist report directly to the Project Manager and are responsible for the implementation of applicable QAPP activities. The Field Engineer and Field Geologist directly supervise

technical studies and work activities in a project task area, and are responsible for assuring that provisions specified in the project implementing procedures are properly executed by qualified personnel. The Field Engineer and Field Geologist are responsible for ensuring that work plans, drawings, and procedures contain qualitative criteria for determining satisfactory work performance and acceptance criteria for determining quality compliance. Due to the scale of this Phase II Site Investigation effort, the Senior Field Professional will also serve as the Site Safety Officer.

Figure B-1 depicts the organization of the project team and identifies individuals responsible for the different roles described above. Table 1 of this QAPP provides proposed primary subcontractors for the proposed work.

Resumes of the key GeoSyntec Consultants project personnel, the Project Principal, Project Manager, Field Engineer, and Field Geologist are included as Appendix C to the Work Plan.

## 4.2 Field Organization and Protocol

### 4.2.1 Field Team Organization

The function of the field team is to carry out the field program outlined in the Work Plan, using an integrated team of scientists, engineers, and other specialists. The field team will be made up of geologists, hydrogeologists, engineers, and/or field technicians. Due to the effort and scope of work involved, team members will have multiple roles. Team members will be assigned as described below:

- Field Engineer and/or Geologist: responsible for directing and coordinating all site activities, day-to-day scheduling of the



field team, ordering equipment and supplies, communicating with the Project Manager, conducting daily briefings, and controlling site access. The Field Engineer and Field Geologist will be responsible for execution of task-specific field activities and supervision of field technicians and subcontractors and will report to the Project Manager. The senior Field Professional will serve as Site Safety Officer as described below.

- Site Health and Safety Officer: responsible for conducting health and safety monitoring of field activities, ensuring that the Site Health and Safety Plan is followed and providing personnel protective equipment. The Site Health and Safety Officer will be assigned by and is accountable to the Project Health and Safety Coordinator. The Site Health and Safety Officer will be the most senior field professional who is assisting with the Phase II Site Investigation.
- Subcontractors: subcontractors including environmental drilling, laboratory analytical work and natural resources studies will be directed by the Field Engineer and Field Geologist.

#### 4.2.2 Start-up Meeting

At the beginning of field activities, a start-up meeting will be held at the site for subcontractors and personnel. The meeting will address the Site Health and Safety Plan, emergency procedures, QAPP, Field Sampling Plan (FSP), decontamination, site access, and ordering of supplies.

#### **4.2.3 Daily Briefings**

At the beginning of each day, the Field Engineer and/or Field Geologist will conduct a briefing with the field team and subcontractors to review the field activities planned for the day, ensure that all field tasks are adequately staffed, and resolve any logistical considerations. The Field Engineer and/or Field Geologist will conduct a safety meeting once each week during one of the daily briefings in his capacity as Site Safety Officer.

#### **4.2.4 Equipment Storage and Supply**

Sampling and monitoring gear, along with health and safety personnel protective equipment, will be stored in the field, in a location to be determined. Personal protective equipment will be ordered by the Site Safety Officer.

#### **4.2.5 Site Communications**

The site may be reached by telephone via the Carus Chemical Company office, (815) 224-6855.

## 5. QUALITY ASSURANCE OBJECTIVES AND DATA QUALITY OBJECTIVES

The main objective of the quality assurance program is to establish procedures to assure precision, accuracy, and completeness in data generated from all Phase II Site Investigation activities, such that these data are scientifically reliable and legally defensible.

Specific QA/QC objectives include:

- ensuring that all samples are collected according to acceptable and recognized procedures;
- ensuring the proper handling and management of samples during and after collection and ensuring that all samples are sufficiently documented to maintain accountable tracking of each sample; and
- ensuring precision, accuracy, and completeness in all laboratory generated data.

The objectives for field analysis measurements are described in the FSP included as Attachment 2 to this QAPP. Precision, accuracy, and completeness procedures for laboratory procedures are provided in Attachment 1 of this QAPP, which presents the ARDL, Inc. Laboratory Quality Assurance Program Plan (LQAPP), hereinafter "ARDL (November, 1992)".

### 5.1 Precision

Precision refers to the level of agreement among repeated measurements of the same parameter. The level of agreement is usually stated in terms of standard deviation, percent difference, or range. The overall precision of each datum is determined by both sampling and analytical factors. Analytical precision is easier to control and

quantify because the laboratory is controlled, and therefore, a measurable environment. Methods of evaluation and of reporting laboratory analytical precision are included in ARDL (November, 1992). Sampling precision is unique to each compound, media, or method, making it more difficult to control and quantify. Data Quality Objectives (DQOs) for precision are defined in the appropriate sections of the FSP.

## 5.2 Accuracy

Accuracy refers to the degree of agreement of a measurement (or an average of measurements of the same parameter) with an accepted reference or true value. Accuracy may be expressed as the difference between the two values, or as the difference converted to a percentage of the reference or true value, or as a ratio. Accuracy is a measure of bias in a system. DQOs for accuracy are defined in the FSP as appropriate. The methods used to report the accuracy of the laboratory analytical work are included in ARDL (November, 1992).

## 5.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations. Sampling rationale is described in Section 4 of the Work Plan and sampling techniques are presented in Sections 3, 4, 5, and 6 of the FSP.

#### 5.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. The ARDL Laboratory will use protocols, methods, detection limits, and custody procedures, which are discussed in ARDL (November, 1992).

#### 5.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units. Sampling techniques described in the Work Plan and FSP are similar to those used during the preliminary site investigation [GeoSyntec, 1992] and the Phase I Site Investigation.

#### 5.6 DQO Levels

Soil, sediment, surface-water, and ground-water samples which will be collected during the Site Investigation will be analyzed for the constituents presented in Table 2 of the Work Plan using methods and detection limits as described in ARDL (November, 1992). The ARDL Laboratory will generate documentation for all samples.

## 6. SAMPLING PROCEDURES

Sampling will be performed in accordance with Section 4 and Tables 1 and 2 of the Work Plan. This section of the QAPP describes the controls to be applied to these activities. The number of samples to be collected will be in accordance with Table 1 provided in the Work Plan.

### 6.1 Field Sampling

The sampling procedures discussed in the FSP (Sections 3, 4, and 5) address the following items as applicable:

- a description of the planned sampling locations;
- a description of the specific sampling procedures to be used;
- a description of containers, procedures, reagents, etc., used for sample collection, preservation, transport and storage;
- a discussion of special conditions for the preparation of sampling equipment and containers to avoid sample contamination;
- a description of sample preservation methods;
- a discussion of the time considerations for shipping samples promptly to the laboratory;
- examples of the custody or chain-of-custody procedures and forms;
- a description of the forms, notebooks, and procedures to be used to record sample history, sampling conditions, and analyses to be performed; and

- a discussion of field QC checks such as field blanks, trip blanks, etc.

## 6.2 Sample Identification

All samples shall be adequately marked for identification from the time of collection and packaging through shipping and storage. Marking shall be on a sample tag or label attached to the sample container. Sample identification shall include, as a minimum:

- project name and code;
- sample identification number;
- sample date and time; and
- initials of the individual performing the sampling (samples for chemical analysis).

Each sample will be assigned a unique identification code to be recorded on the sample tag or label. Each code will be recorded in a sample log and, as applicable, on chain-of-custody documentation. The sample identification codes for all samples collected will function as keys for all field and analytical data that are accumulated.

## 7. SAMPLE CUSTODY

### 7.1 General

Samples will be collected during the Phase II Site Investigation. To accomplish the control of the samples gathered, the following sample chain-of-custody requirements have been established.

### 7.2 Transfer of Custody and Shipment of Field Samples

Field samples will be accompanied by a chain-of-custody record as shown in ARDL (November, 1992). When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody document. This document provides objective evidence of sample custody transfer from the sampler, often through another person, to the analyst in a laboratory. Transfer of custody in the field is the responsibility of the Task Manager or his/her designee.

A sample is under custody if:

- it is in a person's possession; or
- it is in view, after being in possession; or
- it was in possession and then locked up; or
- it is in a designated secure area.

Samples will be packaged properly for shipment and dispatched to the laboratory for analysis, with a separate custody record accompanying each shipping package (one for each package of samples shipped, driven, or otherwise transported to the laboratory).



If samples are shipped to the laboratory by a third party, shipping containers will be padlocked or sealed with a custody seal. The method of shipment, courier name(s), and other pertinent information will be entered in the "Remarks" section on the custody record.

One copy of the chain-of-custody document will accompany the shipment and two copies will be retained; one will be kept in the project files and the other will be sent to the Task Manager.

If sent by mail, the package will be registered with return receipt requested. Freight bills, post office receipts, and bills of lading will be retained as part of the permanent documentation.

### 7.3 Laboratory Custody

Custody at the ARDL Laboratory will be in accordance with ARDL (November, 1992).

## 8. CALIBRATION PROCEDURES AND FREQUENCY

### 8.1 Calibration Identification

Measuring and test equipment used in sampling activities will be uniquely identified by using either the manufacturers' serial numbers, a unique calibration system identification number, or other means. This identification, along with a label indicating when the next calibration is due, will be attached to the equipment. If this not possible, records traceable to the equipment will be readily available for reference.

### 8.2 Calibration Frequency

Measuring and test equipment will be calibrated at prescribed intervals and/or prior to use. Frequency will be based on the type of equipment, inherent stability, manufacturers' recommendations, values given in national standards, intended use, and experience. All sensitive equipment to be used at the project site or in the laboratory will be calibrated or checked prior to use. For field activities, instruments (pH meters, etc.) will be calibrated daily (or more often if readings are suspect). FSP Sections 3 and 4 discusses field instruments for ground-water and surface-water sampling.

It is the responsibility of personnel using measuring and test equipment to check the calibration status from the due-date labels or records prior to using the equipment.

### 8.3 Calibration Standards

Measuring and test equipment will be calibrated, whenever possible, against reference standards having known valid relationships to nationally recognized standards or accepted values of natural physical

constraints. If national standards do not exist, the basis for calibration will be described and documented.

Reference standards will be used only for calibration and will be stored separately from working measuring and test equipment. Equipment that fails calibration or becomes inoperable during use will be removed from service and segregated to prevent inadvertent use, or it will be tagged to indicate it is out of calibration. Such equipment will be repaired and satisfactorily recalibrated.

#### 8.4 Calibration Procedures

EPA approved procedures will be used for calibrating laboratory measuring and test equipment. Calibration of measuring and test equipment may be performed internally using in-house reference standards or externally by agencies or manufacturers.

#### 8.5 Loss of Calibration

If measuring and test equipment is found to be out of calibration, an evaluation will be made and documented to determine the validity of previous measurements made with the equipment. Recalibration of field instruments may be necessary if they are found to be out of calibration. Sampling activity will stop until it has been determined by the appropriate task manager/designee that instruments are operating properly.

## 8.6 Calibration Records

Records will be prepared and maintained for each piece of calibrated measuring and test equipment to indicate that established calibration procedures have been followed.

## **9. ANALYTICAL PROCEDURES**

### **9.1 Laboratory Analysis**

The methods for analyzing ground-water, soil, surface-water and sediment samples for the Carus Chemical Company manufacturing facility are identified in the task specific procedures contained in ARDL (November, 1992). As appropriate, the protocols are methods taken from EPA publications. Actual reference procedures will be maintained in the ARDL laboratory for use by the laboratory personnel.

### **9.2 Field Analysis**

The methods for field analysis of samples are described in Sections 3, 4, and 5 of the FSP.

## **10. DATA REDUCTION, VALIDATION, AND REPORTING**

### **10.1 Data Collection**

Field data will be recorded by field personnel in bound field logbooks and on appropriate standardized data collection forms in ring binders. This will ensure that the same type of measurements or observations are made to provide comparable data at similar locations.

Laboratory data will be collected as described in the ARDL QAPP, associating all laboratory data with the project sample identification code and the laboratory sample identification code.

### **10.2 Data Reduction**

Data reduction will be an on-going activity. Reduction of field data will include raw data collected from project sampling tasks. Laboratory analytical test data will be reduced as described in ARDL (November, 1992).

### **10.3 Data Validation**

Data validation is a systematic procedure of reviewing a body of data against a set of established criteria to provide a specified level of assurance of its validity prior to its intended use. Validation also requires assurance that samples have been properly logged, handled and retained in custody. Checks on sample collection activities will include audits of sample procedures, labeling and logging of samples, handling and transfer of samples to the laboratory and decontamination of sampling equipment. Criteria for data validation will include checks for internal consistency, checks for transmittal errors, checks for verification of laboratory capability, etc. These criteria involve utilization of

techniques such as interpretation of the results of: duplicate sample analysis, spiked addition recoveries, instrument calibrations, detection limits, tests for normality, tests for outliers, and data base entry checks.

#### 10.4 Data Reporting

Field data, analytical data, and data validation reviews will be submitted as a part of all reporting. Analytical results will include results from field and laboratory QC samples.

## 11. INTERNAL QUALITY CONTROL CHECKS

### 11.1 Laboratory

The ARDL laboratory of Mt. Vernon, Illinois, has been selected for performance of all laboratory analyses. The ARDL laboratory is IEPA certified for CLP and will comply with protocols, methods, detection limits and custody procedures, outlined in its QAPP.

The ARDL laboratory QC procedures are detailed in ARDL (November, 1992), and is included in Attachment 1.

### 11.2 Field

Potential sampling, handling, storage, and transportation contamination effects on accuracy will be checked using trip, equipment, and field blanks as specified in Section 1 of the FSP.



## 12. AUDITS

### 12.1 General

To verify compliance with the QAPP requirements detailed herein, the GeoSyntec Consultants' Project Quality Assurance Manager (PQAM) will perform or designate performance of planned and documented audits. These audits will consist of an evaluation of implementing procedures, an evaluation of the scope of work activities and a review of project documentation.

The GeoSyntec Consultants audits will be of two types, namely:

- *Performance Audits*: conducted to verify the laboratory is performing in accordance with ARDL (November, 1992) for a particular method and routine laboratory practices.
- *System Audits*: conducted to verify that field activities which have an impact on data quality are performed in accordance with the QAPP, FSP, and the Work Plan.

The performance audit of the ARDL laboratory will address the laboratory:

- organization and personnel;
- facilities and equipment;
- analytical methodology;
- sample custody procedures;
- quality control; and
- data handling.

System audits will address the scope and quality of the field activities, to include:

- Well installation activities, such as:
  - equipment decontamination;
  - quality of materials used in well installation;
  - well placement technique; and
  - logging/field record keeping.
- Field sampling activities, such as:
  - documentation of activities (log books, etc.);
  - use of proper sampling equipment;
  - proper sample identification;
  - sample preservation;
  - sample packaging;
  - sample shipment; and
  - chain-of-custody.

## 12.2 Audit Schedule

Performance and system audits will be scheduled in a manner to provide coverage coordination with all ongoing activities, scheduled to coincide with the performance of project activities.

Following approval of the Work Plan by IEPA and the development of specific project dates for field and laboratory work, the PQAM will develop an audit schedule which will include the audits to be performed, activities to be audited and the scheduled dates. At a minimum, the Phase II Site Investigation work currently planned will include one system audit and one performance audit.

Initial audits will be performed as early in the task life or work activity as practical.

### 12.3 Audit Preparation

The Project Manager may select and assign a separate Audit Manager who is independent of any direct responsibility for performance of the activities which will be audited. The Audit Manager may select additional audit personnel. Audit personnel will have sufficient authority and organizational freedom to make the audit process meaningful and effective.

The Audit Manager will develop and document an audit plan for each audit. This plan will identify the audit scope, requirements, audit personnel, activities to be audited, organization to be notified, applicable documents, schedule, and written procedures or checklists.

### 12.4 Audit Conduct

Activities that have been selected for audit will be evaluated against specified requirements which will include an objective evaluation of the methodologies, practices, procedures, instructions, and activities. Objective evidence such as documents and records will be examined to the depth necessary to determine if the QA program is effective and properly implemented. Audit results will be documented by auditing personnel, analyzed by the Project Manager, and reported to Task Managers having responsibility for the area audited. These managers will review and assess the results and take appropriate action.

### 12.5 Audit Reporting

Audit reports, signed by the Project Manager, will be issued and will include the following information, as appropriate:

- description of the audit scope;

- identification of the auditors;
- identification of persons contacted during audit activities;
- summary of audit results, including the effectiveness of the QA program elements which were audited; and
- description of each reported audit finding in sufficient detail to enable corrective action to be taken by the audited organization if necessary.

The GeoSyntec Consultants' Project Manager will investigate audit findings, determine the cause of any adverse condition identified in the findings, schedule corrective action (including measures to prevent recurrence), evaluate the impact of the finding on completed work, and prepare a written report of action taken or planned. The adequacy of audit responses will be evaluated by the Project Manager.

A tracking plan for audit findings will be established to help assure that all findings are appropriately addressed and to identify trends in significant conditions that are adverse to quality. Follow-up action, including re-audit of deficient areas, will be taken as necessary to verify whether corrective action is accomplished as scheduled.

### 13. PREVENTIVE MAINTENANCE

#### 13.1 General

Periodic preventive maintenance is required for all sensitive field and laboratory equipment. Field instrument manuals will be kept on file in the field office for reference should equipment need repair.

Field equipment requiring routine maintenance will be tagged with maintenance labels indicating the date of required maintenance, the person maintaining the equipment, and the next maintenance date. Information pertaining to life histories of field equipment maintenance will be kept in individual equipment history logs with each instrument.

Preventive maintenance procedures for the laboratory equipment are detailed in ARDL (November, 1992), Attachment 1 to this Appendix.

## **14. DATA MEASUREMENT ASSESSMENT**

### **14.1 Precision and Accuracy**

Precision and accuracy are overall indicators of data quality. As with data quality and quantity, precision and accuracy assessment procedures are compound, media, and method-specific and are specified and described in the appropriate FSP procedure or sampling and analysis plan.

The historical precision and accuracy, if achieved by different analytical techniques, will be reviewed for each task to allow a comparison of the analytical techniques. In addition, representativeness, completeness, and comparability will be reviewed and addressed.

### **14.2 Data Assessment and Completeness**

Following validation, data will be assessed to determine its representativeness and completeness. The actual sampling techniques used by field personnel will be reviewed by the Task Manager for deviations from the written sampling procedures. If deviations are found, their impact on the representativeness of data will be assessed.

Any lost or suspect data will be evaluated in terms of sample location, analytical data lost (fraction, individual parameter, etc.), decision to be made with the data, and risk associated with an erroneous decision. Critical locations and critical analytical data will be examined to determine their adequacy and impact on the overall objectives of the project.

## 15. CORRECTIVE ACTION

### 15.1 Corrective Action

Nonconformances identified in either QA audits or routine quality control will be evaluated and the need for corrective actions determined. Upon receipt of notification that corrective action is required, the affected task manager or organization(s) will:

- assure that immediate action is taken to correct the nonconforming condition;
- determine the cause of the nonconforming condition;
- assure that controls have been reviewed, implemented, monitored, and revised, if necessary; and
- report all actions taken to project management, including quality assurance.

### 15.2 Verification

Effectiveness of corrective action will be verified by the Project Manager and monitored at periodic intervals to ensure that corrective action is continuing in its application.

**16. QUALITY ASSURANCE REPORTS TO MANAGEMENT**

The Audit Manager will prepare a report to the Project Manager on the results of each quality assurance audit. The report will address separate quality assurance sections to include data quality information collected during the project.



TABLE 1

PRIMARY SUBCONTRACTOR SUPPORT  
SITE INVESTIGATION

<u>Subcontractor</u>	<u>Contact</u>	<u>Task Assignment</u>
Layne Environmental Services W229 N 5005 Du Plainville Road Pewaukee, Wisconsin 53072	Amy Pomeroy (414) 246-4646 fax (414) 246-4784	Environmental Drilling Tasks 3 and 7
ARDL, Inc. P.O. Box 1566 1801 Forest Street Mt. Vernon, Illinois 62864	Dr. L.V. Gibbons (618) 244-3236 (800) 842-7134 fax (618) 244-1149	Certified Illinois Analytical Laboratory - Task 12
CIH Services 1520 Sundale Drive Lawrenceville, Georgia 30245	Paul Sawyer, C.I.H. (404) 822-1900	Consulting Health and Safety Services

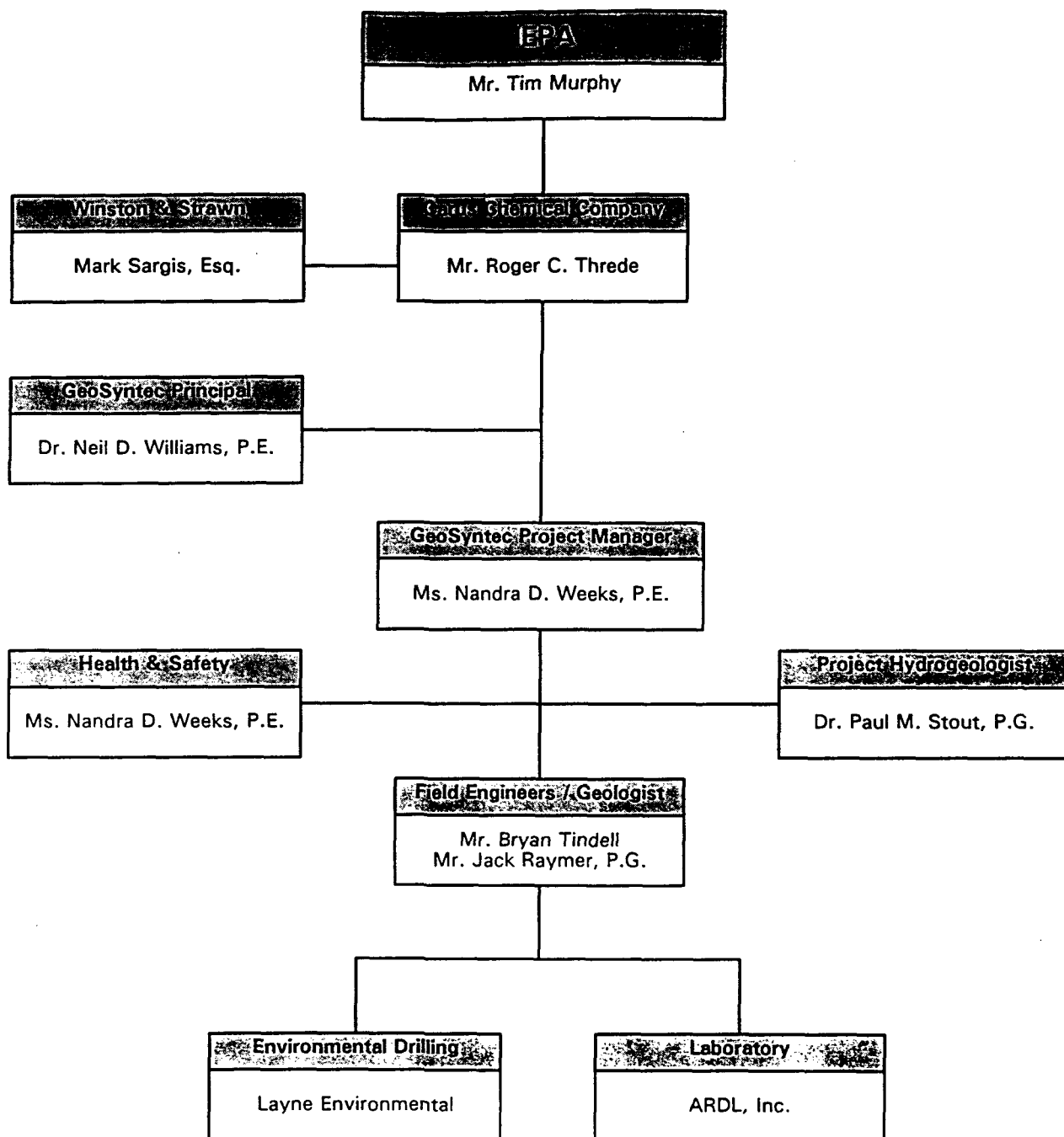


FIGURE 1: Project Team Organization



GEOSYNTEC CONSULTANTS

Figure No. B-1  
Project No. FE2167  
Document No.

**ATTACHMENT 1**

**LABORATORY QUALITY ASSURANCE PROJECT PLAN**

# QUALITY ASSURANCE PROGRAM PLAN

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Approved By:

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Date

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President and Laboratory Director

11/25/92

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11/25/92

D.J. Gillespie  
Technical Services Manager

11/25/92

QUALITY ASSURANCE

PROGRAM PLAN



ARDL, Inc.

CHEMISTRY — BIOLOGY — PHYSIOLOGY — ENGINEERING  
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## INTRODUCTION

This document is the Quality Assurance Program Plan (QAPP) prepared by ARDL, Inc., Mt. Vernon, Illinois. The document contains a description of the basic approach taken by ARDL, Inc. to assure that the data produced in the laboratory are reliable and of the highest possible quality. The main body of the text (Sections 1 through 14) and the Standard Operating Procedures and other supporting information in the Appendix provide details regarding: 1) the organization of ARDL's analytical laboratories; 2) the methods and procedures utilized for sampling and analysis; 3) procedures relative to data reduction, validation and reporting; 4) quality control and quality assurance procedures and 5) other matters pertaining to operations and management that effect data quality and reliability.

As indicated in the text, all analytical procedures and methods referenced herein are taken from the following sources:

1. "Test Methods for Evaluating Solid Waste: Physical and Chemical Methods", SW-846, 3rd Edition (Revisions 0 through 2), USEPA, November, 1986.
2. "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, USEPA, March, 1983.
3. "Standard Methods for Examination of Water and Wastewater", 18th Edition, APHA/AWWA/WEF, 1992.
4. USEPA CLP protocols as described in ILM02.0 (SOW for Inorganic Analysis) and ILM01.0 through ILM01.7 (SOW for Organic Analysis).

**ORGANIZATION AND RESPONSIBILITY**

ARDL is organized along conventional lines as indicated by Figure 1. Dr. L.V. Gibbons is President and Laboratory Director and all personnel in the laboratory engaged in analytical work either report directly to him or through the Manager of Technical Services. As shown in Figure 1, the General Laboratory Manager acts independently as the administrator of ARDL's quality control program. The duties of key personnel involved in analytical work are summarized below.

## 1.1

**General Laboratory Manager**

Responsible to the Laboratory Director for efficiency and productivity in all aspects of technical operations in the laboratory. Activities that impinge directly on ARDL's analytical efforts are as follows:

- Review of project status to insure that requirements are being met and that the responses to customer needs are timely.
- Assess modifications in standard analytical methodologies and/or customer needs which may occur from time to time and identify, approve and coordinate changes in operating procedures, staff or equipment needed to meet those new requirements.
- Evaluate laboratory facilities and capabilities and assess recommendations for improvements which may enhance responses to customer needs.
- Monitor manpower loadings to insure adequate staffing of the analytical effort and correct deficiencies which may occur by approval of personnel reassignments from other laboratory duties or hiring of new staff.

## 1.2

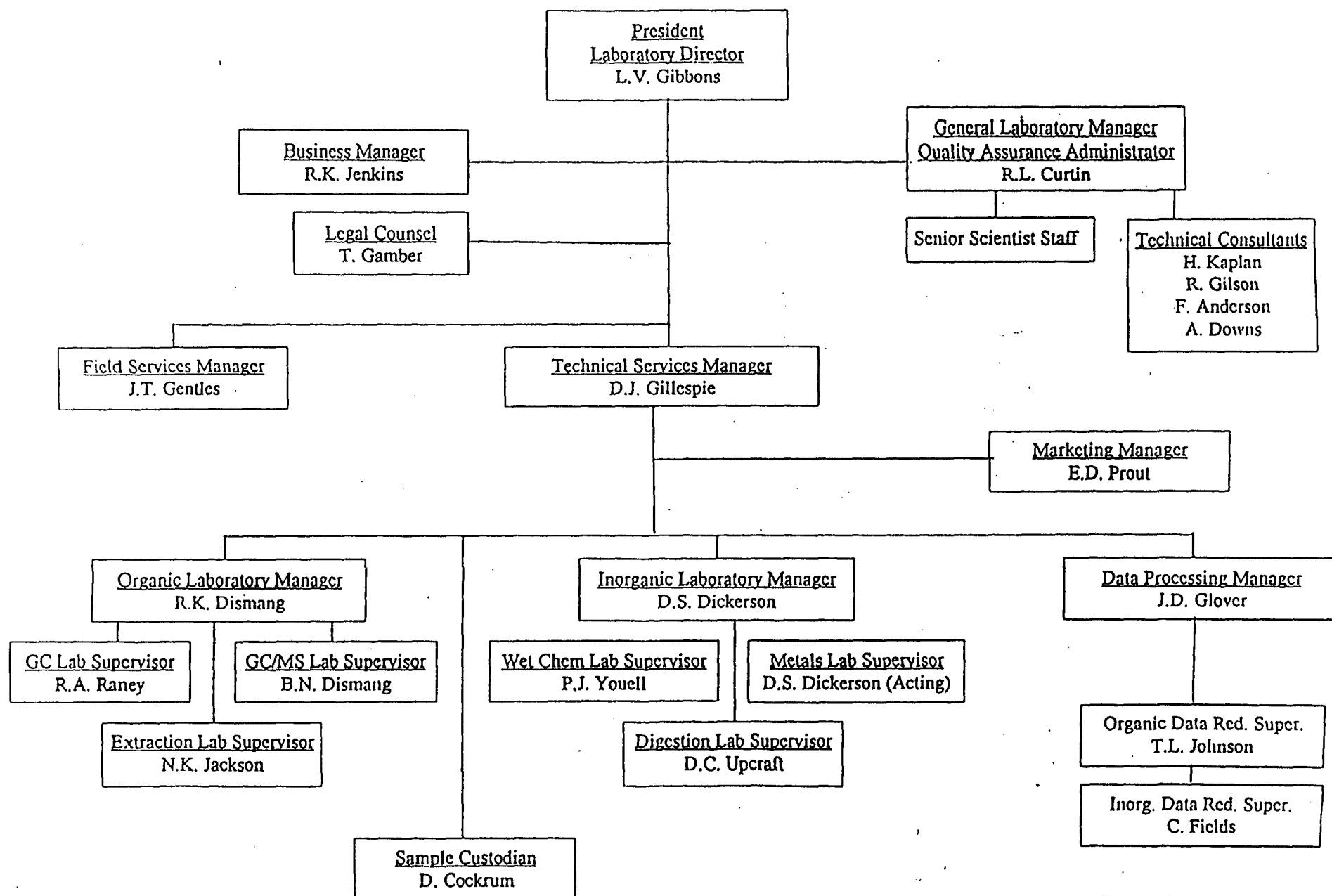
**Technical Services Manager**

Responsible to the Laboratory Director for safe, efficient and timely operations in the analytical laboratory. Some of the specific tasks involved in that effort are:

- Monitor work progress on a daily basis to insure that samples are being handled in accordance with customer requirements in a timely fashion.
- Coordinate efforts of the Inorganic and Organic Laboratory Managers to insure adequate staffing and efficient scheduling to meet customer needs.
- Monitor laboratory facilities and capabilities and make recommendations for improvements which may enhance response to customer requirements.

FIGURE 1

ARDL, INC. OPERATING ORGANIZATIONAL CHART



DATE: August 1992

Figure 1A

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- Evaluate changes in requirements which may result from modifications in standard analytical methodologies or new needs of customers and make recommendations for changes in staff, equipment or procedures which may be needed to meet those new requirements.
- Coordinate with the Inorganic Laboratory Manager, Organic Laboratory Manager and Quality Assurance Administrator relative to adherence to provisions of the Quality Assurance Program Plan (QAPP).
- Approval of finished data for release to the customer.

### 1.3 Inorganic Laboratory Manager

Responsible to the Technical Services Manager for the safe, efficient operation of laboratory facilities engaged in sample analysis for metals, anions, BOD/COD, solids, certain organic moieties and other parameters associated with water/waste water quality. Specific activities relative to that effort are as follows:

- Daily supervision of analyst activities including review of results for completeness and accuracy, analysis scheduling, analyst assignments, consultation with analysts regarding special problems, etc.
- Monitor analyst adherence to QC requirements.
- Coordinate with the Technical Services Manager and Quality Assurance Administrator regarding overall adherence to provisions of the QAPP and identify corrective actions where required.
- Approve requisitions for materials.
- Supervise training of personnel in analytical procedures.
- Prepare laboratory reports for approval by the Technical Services Manager.
- Review preventative maintenance logbooks and coordinate service work on instruments as required.

### 1.4 Organic Laboratory Manager

- Responsible to the Technical Services Manager for the safe, efficient operation of laboratory facilities involved in gas chromatographic analyses. Specific activities relative to that effort are as follows:

- Daily supervision of analyst activities including review of results for completeness and accuracy, sample preparation and analysis scheduling,



consultation with analysts relative to special problems, analyst assignments, etc.

- Monitor analyst adherence to QC procedures.
- Coordinate with the Technical Services Manager and Quality Assurance Administrator regarding overall adherence to provisions of the QAPP and identify corrective actions where required.
- Approve requisitions for materials.
- Supervise training of personnel in analytical procedures.
- Operate or supervise operation of the GC/MS system on a daily basis and monitor the results obtained to insure adequate function.
- Prepare laboratory reports for approval by the Technical Services Manager.
- Review preventative maintenance logbooks and coordinate service work on instruments as required.

#### 1.5 Quality Assurance Administrator

Responsible to the Laboratory Director for administration of the QAPP. Specific activities relating to that effort are as follows:

- Review of analyses in progress to identify the pertinent requirements necessary to fulfill the specifications and guidelines to the QAPP.
- Review of analytical results obtained on QC samples.
- Advise the Inorganic and Organic Laboratory Managers relative to the acceptability of QC data and coordinate with those individuals in cases where QC data is outside acceptable limits so that appropriate corrective actions may be taken.
- Supervise maintenance of QC logbooks and records on a continuous basis.
- Report to the Laboratory Director regarding QAPP status on a regular and frequent basis.

#### 1.6 Analysts

Responsible to either the Inorganic or Organic Laboratory Managers, as appropriate, for safe, efficient performance of analytical procedures. Specific activities relating to those efforts include:

- Performance of assigned analytical procedures in the prescribed manner.
- Performance of the required QC procedures and analyses.
- Maintenance of laboratory notebooks and log books.
- Preparation of requisitions for materials.
- Performance of preventative maintenance on instruments and equipment associated with assigned analyses.

## 2.0 DEFINITIONS AND QUALITY ASSURANCE OBJECTIVES

An effective quality assurance program for an analytical laboratory must address means for achieving and maintaining acceptable precision, accuracy, completeness, representativeness and comparability of the data produced as a result of work performed in that laboratory. For purposes of this discussion these elements are defined as follows:

### 2.1 Definitions

#### 2.1.1 Precision

Precision is the degree of mutual agreement among individual measurements (replicate or repetitive) made under prescribed conditions with a single test procedure.

Precision is measured by determining the agreement between sample duplicates expressed in terms of relative percent difference (RPD). RPD is calculated by the equation:

$$RPD = (S - D) / ((S + D) / 2) \times 100$$

Where;

S = sample concentration

D = duplicate concentration

Control limits of for RPD appear in subsequent sections of this document.

#### 2.1.2 Accuracy

Accuracy is the difference between an average value and the true value when the latter is known or assumed.

Percent recovery of laboratory control samples and spikes are used to assess accuracy. Percent recovery (%R) is calculated by one of the following relationships:

For Laboratory Controls:  $\%R = 100 (\text{observed}/\text{known})$ , or

For Spikes:  $\%R = 100 ((\text{observed}-\text{background})/\text{spike})$

Recovery limits for spikes and laboratory control samples appear in subsequent sections of this document.

#### 2.1.3 Completeness

Completeness is the percentage of valid data generated by an analytical system. ARDL's goal is to provide acceptable valid data for ninety percent (90%) or more of the

determinations required by the customer. The procedures described in this document were developed to meet that goal.

#### **2.1.4      Representativeness**

Representativeness is the degree to which the data accurately and precisely represent the sample source or site as determined by the data user and sampling team.

#### **2.1.5      Comparability**

Comparability is the measure of the confidence with which one set of analytical results from a source or site can be compared to another set, as determined by the data user and sampling team.

#### **2.2        Quality Assurance Objectives**

A program by which to gauge the acceptability of these factors in data generated in the laboratory has been developed and implemented. The objectives of that program are summarized below:

1. To provide data for measurement of the accuracy, precision and completeness of analytical results obtained in the laboratory.
2. To provide permanent records of instrument performance as a basis of validating data and projecting repair or replacement needs.
3. To upgrade the overall quality of laboratory performance.
4. To help ensure that the analytical work will withstand legal scrutiny in regulatory actions.
5. To improve record keeping and report writing.
6. To help ensure sample integrity.
7. To help detect training needs within an analytical group.
8. To identify weak methodology.

Detailed explanations and general guidelines for meeting these objectives are developed in Sections 7.0 (Quality Control Requirements), 8.0 (Data Handling and Reporting), 9.0 (Internal Quality Control Checks and Frequency), 12.0 (Specific Procedures to Assess Quality Assurance Data) and 14.0 (Quality Assurance Reports of Management) of this document. As the needs of the customer dictate, these procedures may be augmented by additional efforts and this document amended by attachment of additional standard operating procedures to satisfy those requirements.

### 3.0 SAMPLE HANDLING

#### 3.1 Sample Containers, Preservations and Holding Times

Specifications for sample containers and preservatives are tabulated in Appendix C. Containers meeting these requirements may be prepared by the customer or obtained from appropriate vendors. These containers may be obtained from ARDL if prior arrangements to that effect are made.

ARDL obtains precleaned sample containers from outside vendors and also prepares those containers in the laboratory. The vendors from whom these containers are purchased certify that the containers have been prepared in accordance with applicable USEPA protocols. The procedures employed by ARDL personnel when preparing the containers in the laboratory are those specified by USEPA protocols.

Specifications for holding times used by ARDL are taken from references 1 and 2 cited in the introduction of this document. The holding times before analysis and/or preparation for analysis used by ARDL are listed in Appendix C. Specifications for storage conditions for the raw sample and all extracts or digestates also appear in Appendix C. If samples are received which require analyses not listed in Appendix C, the holding times listed in these references will be utilized.

#### 3.2 Sample Retention and Disposal

After analysis is complete on a sample set the samples and/or sample processing products will be held at 4°C for 30 days. After that time the samples are held for an additional 90 days. the samples may or may not be refrigerated during this additional 90 day period. After this 120 day period has elapsed ARDL returns the samples to the customer unless specific arrangements have been made to the contrary. When ARDL is charged with disposal of samples, that disposal will be accomplished in complete accordance with all regulations governing such activity.

Samples being held for disposal are tracked on a database integral to the computerized log-in system in use in the laboratory. Dates for disposal are system generated when all analytical work is complete and the sample group files are archived. The database can be searched by date of receipt, date of analysis completion, customer and other variables to determine which samples are due for disposal. As samples are selected for disposal by the operator, the entries are purged from the database and a record of the disposal is prepared. That record is retained in laboratory files indefinitely.

## **4.0            CHAIN-OF-CUSTODY**

### **4.1            Custody Definition**

For purposes of this document sample custody is defined as: 1) awareness of sample location; 2) specific knowledge of the individuals who currently have or have had possession of the sample; and 3) retention of the materials in a manner which insures that only individuals authorized to work with sample materials have access to them; and 4) development of records which demonstrate that sample integrity has been maintained.

### **4.2            Transfer of Custody in Shipment**

Samples may be collected either by customer personnel or by ARDL employees. When collection is performed by the customer the samples are usually delivered to ARDL by common carrier but may, in some instances, be hand carried to the laboratory. On delivery a Chain-of-Custody Form and an analysis request form are prepared (see Appendix B for exemplars of the forms in current use by ARDL).

The ARDL sample receipt clerk (or designated alternate) is authorized to accept samples and is charged with the responsibility for proper completion of the required sample receipt documentation. As required, analysts from the inorganic and organic laboratories are assigned to assist the sample receipt check in sample log-in procedures.

When samples are collected by ARDL personnel, chain-of-custody and analysis request documentation is prepared at the time of sample custody transfer at the customer's facility. That documentation is returned to ARDL along with the samples.

In all cases, chain-of-custody and analytical request documents become part of the permanent file relative to the samples collected. Those files are retained indefinitely in ARDL's facility.

### **4.3            Laboratory Custody Procedures**

All samples in storage at ARDL except those intended for volatiles analysis are retained in custody of the Sample Custodian until released as required for analytical work. The samples are kept in locked, walk-in refrigerators located in close proximity to the log-in work area. Keys to these refrigerators are held by the Sample Custodian. As directed by their supervisors, analysts obtain the keys to obtain materials for analysis. A record of the custody change is made by the analyst and checked by the Sample Custodian at the time the samples are taken from general storage. The quantities used for each analysis are traceable through dated entries made in analyst laboratory notebooks. If the samples will be depleted by the analytical procedures planned, the record contains a note to that effect. When samples are returned by the analysts a record of the custody change is made. Internal custody records are retained indefinitely in laboratory files.

Custody for samples intended for volatiles analysis is transferred to the Organic Laboratory Manager immediately after log-in procedures are complete. A record of the custody change is made and retained indefinitely in laboratory files. These samples are taken to the volatiles laboratory where they are stored in conventional refrigerators until analyzed. As with all other samples, the quantities taken for analysis are traceable through logbook entries. Like all of ARDL's facility, access to the volatiles laboratory spaces is restricted to ARDL employees and escorted visitors.

#### 4.4 Laboratory Security

All outside entrances to ARDL are locked during normal working hours as well as after hours. Access to the facility by employees during the day is possible through three doors fitted with Hirsch scramble pads/card readers and magnetic locks. Each employee is issued a keypad code which is used for entry/exit and movement through control doors within the facility. Individual employee codes may have different levels of access within the building based on time of day, day of week, etc.

Access to the facility by non-employees is permitted through a front and a rear entrance. The rear entrance is located in shipping and receiving and is monitored by the Shipping/Receiving Clerk. Access to the facility through the front entrance is monitored by the receptionist. All visitors must sign the visitor's log and are issued a visitor's card which must be used for entry through control doors. All visitors are escorted while within the ARDL facility.

The access control system described above is connected to a computer system which logs all activity through each door. Data as to person entering, time of entry/exit, code used, etc. are readily available for onscreen review. Daily review of all logs and printouts of facility utilization by employee/visitor is conducted.

After hours the entire facility is protected by an electronic alarm system. The system consists of perimeter sensors mounted on all windows and outside doors and internal motion and infrared detector sensors. The doors to the refrigerated sample storage areas and doorways to several internal areas where confidential information is stored are connected to the alarm system. Unauthorized entry to facility causes an alarm to sound at local police headquarters as well as on-site.

Police response is typically 5 to 10 minutes after an alarm is received. After dispatch of an officer, the police notify a member of the senior management staff. In the company of the responding officer, that individual inspects the facility. Corrective actions are taken as appropriate when the inspection is completed and the alarm system is brought to readiness and reset.

## 5.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments in use at ARDL for sample preparation and analysis are subject to routine calibration procedures prior to acquisition of sample data. The procedures utilized on instruments and equipment associated with determining the concentrations of the various target analytes of routine interest are described below. In general these procedures conform to guidelines given in 40 CFR 136 and SW-846.

### 5.1 Balances

Calibration procedures for balances are fully described in Appendix H.1.

### 5.2 pH Meter

Standard pH meters with temperature compensating capabilities and fitted with gel-filled combination electrodes are employed for pH measurements at ARDL. When not in use, electrodes are kept immersed in pH 7 standard buffer.

Meter and electrode function are checked daily with standard buffers at pH 4.0, 7.0 and 10.0. After determining the temperature of the solutions, adjustments of front panel controls are made as required so that the instrument reads pH 7.0 in the pH 7.0 buffer. After this adjustment, the readings obtained at either end of calibration range must be within  $\pm 0.2$  pH units of true value for the buffer involved.

The readings obtained are recorded in a laboratory logbook maintained for the purpose. If the criteria are not met, the meter is removed from service and appropriate corrective action is taken to restore proper operation.

### 5.3 Spectrophotometers

In general, the methods in use for atomic absorption, plasma and light spectrophotometric analysis at ARDL are taken from references 1 and 2 cited in the introduction to this document. The text below details the procedures in use for such analyses on a daily basis.

When CLP protocol is required for a sample set, the procedures and specifications given in the most recent SOW for Inorganic Analysis are followed. CLP procedures are not detailed in the Sections below.

#### 5.3.1 Initial Calibration

##### 5.3.1.1 Setup and Standards Preparation

The instrument is activated and an appropriate interval allowed for warmup. As appropriate, instrument zero, full-scale deflection and preliminary adjustments are made in accordance with the manufacturers instructions. Once adjusted, the instrument is ready for calibration using appropriate standards. If an instrument does not respond as required during



initial setup, it is removed from service and appropriate corrective action taken to restore proper function.

For analyses involving atomic absorption spectrophotometry four or five point calibrations consisting of a blank and the requisite number of standards are usually employed by ARDL. Six point calibrations consisting of a blank and five standards are employed for analyses involving visible light spectrophotometers.

As described below, working standards for both initial calibration and continuing calibration procedures are prepared fresh daily from stock solutions or by weighing appropriate quantities of reagent chemical and diluting to volume with the required solvent. All stock standard solutions are assigned unique code numbers and details relative to their origin and preparation are recorded in a standards logbook as described in Appendix A.1, Standard Operating Procedure, Preparation of Standard Solutions.

To prepare working standards the analyst identifies the stock utilized by vendor lot or stock ID number and records the quantities taken and the final volume to which those amounts were diluted. For all analyses calibration standards are prepared at systematically increasing concentrations near the contract required detection limit (CRDL) and the top of the linear range for the instrument involved. ARDL utilizes the CRDL values given in most current revision of the USEPA Inorganic SOW referenced above. An inorganic target compound list and the CRDL's for those analytes taken from that document appear in Appendix F.1.

#### 5.3.1.2 Curve Generation

The absorbance or transmittance of the standards is determined on the instrument. For instruments without an associated data system, the readings observed are recorded in the analyst's logbook, in such cases appropriate notations regarding the identity, source and concentration of each standard solution are also made in the logbook. ARDL logbooks are retained on file indefinitely. In cases when full CLP documentation is required for a sample set photocopies of logbook pages showing the calibration data are submitted to the customer as part of the completed data package.

For instruments with an attached data system the calibration data are typically in hard copy as the readings are made. The original of this printout is retained on file indefinitely in the laboratory. In cases when full CLP documentation is required for a sample set photocopies of logbook pages showing the calibration data are submitted to the customer as part of the completed data package.

The linearity of the standard curve is evaluated by calculation of the regression coefficient,  $r$ . Linearity is assumed if the value for  $r$  equals or exceeds 0.995. If the curve proves to be linear, sample concentrations and required QC values are calculated using the regression equation. In those cases where a nonlinear curve is expected based on the method employed, the data are plotted and sample concentrations read directly from that plot.

If a nonlinear curve is obtained where the method indicates that linearity is expected, appropriate corrective action is taken and data for a new standard curve are acquired.

### **5.3.2 Calibration Verification**

Once sample analysis begins the calibration of the analytical system is verified at least every ten samples and at the end of the run. Calibration verification consists of two parts: 1) analysis of a standard containing a known concentration of analyte; and 2) analysis of a blank which contains none of the analyte. Calibration verification is performed immediately after initial calibration procedures are complete and immediately after analysis of every tenth sample.

#### **5.3.2.1 Initial Calibration Verification**

Immediately after preparation, the calibration curve is verified by analysis of 1) an aqueous check standard (ICV) prepared from material of known concentration which is not the same as the standards used in development of the standard curve, and 2) a blank of reagent water (ICB) which contains approximately the same concentration of acid(s) as the check standard.

Results observed for the ICB must be within  $\pm$  CRDL for the analyte involved. Results observed for the ICV must be within 20% of true value.

When the measurements on the ICV or ICB exceed control limits, the analysis is terminated and appropriate corrective action is taken. Once the problem is resolved, the initial calibration procedure described in Section 5.3.1, above is repeated in its entirety before sample analysis can proceed.

#### **5.3.2.2 Continuing Calibration Verification**

After analysis of every tenth sample, calibration of the instrument is verified by evaluation of, 1) a standard (CCV) prepared at a concentration near the middle of the initial calibration range, and 2) a blank (CCB). In practice the CCV and CCB are the same solutions used for the initial calibration verification procedure described above. The source of the standard and the blank, however, may be any source of known concentration.

Results observed for the CCB must be within  $\pm$  CRDL for the analyte involved. Results observed for the CCV must be within 20% of true value.

When the measurements on the CCV or the CCB exceed control limits, sample analysis is suspended and appropriate corrective action is taken. At the option of the analyst, reanalysis of the CCV or the CCB to confirm that a problem has been encountered is permitted as a first corrective action. If that reanalysis shows that the first evaluation was in error, sample analysis can be continued provided that; 1) recovery of the midrange initial calibration standard is within 10% of true value, and 2) the frequency of the continuing calibration verification procedure is doubled over the next 10 samples.

If the optional first corrective action confirms that the instrument has lost calibration or if the analyst elects to bypass that step, additional corrective actions are taken as appropriate (see Section 11, below). Once the problem is resolved, the initial calibration procedure described in Section 5.3.1, above is repeated in its entirety. Any samples evaluated since the last compliant calibration point are reanalyzed.

#### **5.4            Inductively Coupled Argon Plasma (ICAP)**

##### **5.4.1        Initial Calibration**

##### **5.4.1.1     Setup and Standards Preparation**

The plasma is energized in accordance with the manufacturers specifications and the instrument allowed to stabilize over an interval of at least twenty minutes. After stable conditions are achieved, the instrument is profiled using the mercury reference lamp. The system reference to the mercury line at 254.800 nm must be within +/- 0.3 scan units of true. If that specification is not satisfied, manual adjustment is made in accordance with the manufacturer's instructions until the required value is obtained.

Working standards are prepared as required from stock solutions. Preparation of these standards is fully documented as described above for spectrophotometers (Section 5.3.1.1).

A two point curve is required for initial calibration of this instrument. The curve is established by analysis of a blank and a series of standards containing the target analytes in a range of concentrations.

##### **5.4.1.2     Curve Generation**

The blank and standard solutions are introduced into the instrument and the readings obtained are retained by the data system. Linearity is assumed for all elements between the lower limit of IDL (see Section 5.4.1.4, below) and an upper limit established quarterly (see Section 9.0, below) for each analyte.

The calibration data are typically printed out in hard copy just after each set of instrument readings is made. The printout is stamped by the data system with the date and time when the calibration was performed. The original of this printout is retained on file indefinitely in the laboratory.

#### **5.4.2        Calibration Verification**

Calibration is verified immediately after completion of the initial calibration procedure and after analysis of every tenth sample. Calibration verification consists of two parts: 1) analysis of a standard containing a known concentration of analyte(s); and 2) analysis of a blank which contains none of the analyte(s).

#### 5.4.2.1 Initial Calibration Verification

Immediately after completing initial calibration of the instrument, the calibration curve is verified by analysis of 1) a series of aqueous check standards (ICV) prepared from materials of known concentration which are not the same as the material used for development of the standard curve, and 2) a blank of reagent water (ICB) which contains approximately the same concentration of acid(s) as the check standard.

Results observed for the ICB must be within  $\pm$  CRDL for the analytes involved. In those cases where the IDL exceeds CRDL (e.g., lead by ICP), the results observed for the ICB must be  $\pm$ IDL. Results observed for the ICV must be within 10% of true value.

If one or more analytes fail the acceptance criterion, the analyst may elect to continue the run at his discretion. The data obtained subsequently during the run, however, are valid only for those analytes for which the initial calibration has been verified.

For analytes which fail the initial calibration verification criteria, all subsequent data are invalid. The initial calibration procedure described in Section 5.4.2.1 must be repeated in its entirety for these analytes.

#### 5.3.2.2 Continuing Calibration Verification

After analysis of every tenth sample, calibration of the instrument is verified using a series of aqueous check standards of known concentration and a blank. In general practice these solutions are the same as those used for ICV's and ICB.

Results observed for the CCB must be within  $\pm$  CRDL for the analytes involved. In those cases where the IDL exceeds CRDL (e.g., lead by ICP), the results observed for the CCB must be  $\pm$ IDL. Results observed for the CCV's must be within 10% of true value.

If one or more analytes fail the acceptance criterion, sample analysis is suspended. At the option of the analyst, reanalysis of the CCV or CCB is permitted as a first corrective action to confirm that a problem has been encountered. If this preliminary step shows that the first evaluation was in error, sample analysis may be continued provided; 1) analysis of the initial calibration standard is within 5% of true value, and 2) that continuing calibration verification is performed at least twice during analysis of the following ten samples.

If the optional first corrective action confirms that calibration has been lost or if the analyst chooses to bypass that step, two paths may be followed. The run may be continued to acquire data for analytes which remain in calibration or, the run may be terminated and appropriate corrective actions taken to restore calibration for all analytes. If the run is continued, the data obtained subsequently are valid only for those analytes for which continuing calibration has been verified. If the run is terminated and other corrective actions taken (see Section 11, below), the initial calibration procedure outlined in Section 5.2.4, above, is repeated in its entirety.

### 5.4.3

#### Interference Check Sample

Analysis of interference check standards (ICS solutions; ICSA and ICSAB) is performed at the beginning and end of each analytical run or twice during each eight hour shift, whichever is more frequent, to verify interelemental and background correction factors. ICSA contains the interferences aluminum, calcium, iron and magnesium at approximate concentrations of 500, 500, 200 and 500 mg/L, respectively. ICSAB tabulated below at the approximate concentrations noted.

<u>Analyte</u>	<u>Concentration (mg/L)</u>
Silver	1.0
Barium	0.5
Beryllium	0.5
Cadmium	1.0
Cobalt	0.5
Chromium	0.5
Copper	0.5
Manganese	0.5
Nickel	1.0
Lead	1.0
Vanadium	0.5
Zinc	1.0

Analysis of the ICS at the beginning of a run immediately follows analysis of the last initial calibration standard (ICV and ICB prior to ICSA/B). Analysis of the ICS at the end of a run immediately follows analysis of the last sample or QC solution of interest to the customer. If the ICS are analyzed during a run to satisfy the eight hour frequency requirement, the sequence of solutions analyzed just before or just after is immaterial.

ICS solutions are either purchased as certified standards or prepared in the laboratory using certified solutions of the analytes involved. True values for purchased solutions are taken from the certified values obtained from the vendor. The true values for the levels of the analytes in laboratory prepared ICAS are established statistically in at least three valid analytical runs in which the analysis of ICSA/ICSAB solutions of known concentrations satisfy criteria for all of the elements listed above.

The advisory limits of recovery for non-interferent analytes in ICSAB solutions are 80 - 120% of true value. If recovery falls outside of these limits no immediately corrective actions are required.

### 5.5

#### GC/MS

In general all performance checks, calibrations, calibration verifications and other QC data requirements relative to GC/MS analyses meet the criteria given the most recent USEPA document entitled Statement of Work for Organics Analysis. These requirements are somewhat

more demanding than those outlined in "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", SW-846, 3rd Ed., USEPA, November 1986; or 40 CFR Part 136, Federal Register, October 26, 1984 and have been adopted by ARDL as part of a continuing effort to improve data quality.

#### **5.5.1      Instrument Setup and Standards Preparation**

Unless shut down for maintenance, all GC/MS systems at ARDL remain in readiness to begin the calibration sequence. As a first step in that sequence, calibration gas (perfluorotributyl-amine) is introduced directly into the mass detector in a manner specified by the manufacturer. The voltage settings on the detector elements are adjusted either manually or by use of software routines so that the ion abundances obtained at the detector output are within limits specified by the manufacturer. When the abundances observed meet these specifications, maximum detector performance has been achieved and preliminary instrument setup is complete. If unsatisfactory results are obtained in this test, the instrument is removed from service and appropriate corrective action is taken to restore proper function.

As already described, working standards for both initial calibration and continuing calibration procedures are prepared from stock solutions or by weighing appropriate quantities of reagent chemical and diluting to volume with the required solvent. All stock standard solutions are assigned unique code numbers and details relative to their origin and preparation are recorded in a standards logbook as described in Appendix A.1, Standard Operating Procedure, Preparation of Standard Solutions. Details of the steps involved in preparation of standards for calibration of GC/MS instruments are fully described in Appendix A.3, Standard Operating Procedure, Preparation of Standards for GC/MS.

#### **5.5.2      System Performance Verification (Tuning)**

System performance is verified by evaluating the mass spectrum obtained by direct injection of either p-bromofluorobenzene (BFB) or decafluorotriphenylphosphine (DFTPP) with the specifications appearing in Appendix C.1 and C.2, respectively. If calibration for analysis of volatile organics is intended, the compound injected and analyzed is BFB. If calibration for semivolatile analysis is intended, the compound injected and analyzed is DFTPP.

If the criteria appearing in the referenced tables are satisfied, calibration of the instrument as described in Section 5.5.3 below may proceed. If the criteria appearing in the referenced tables are not met, the instrument is removed from service and appropriate corrective action taken to restore proper function.

Verification of system performance is valid for a period of exactly twelve (12) hours from the time of injection of the performance check compound. Analytical samples (calibration standards, samples and/or QC check solutions) may be introduced into the instrument system at any time within that interval.

At the end of the twelve hour period, performance is reverified as described in this section and the system is recalibrated using the procedures described below. The preliminary

steps for achieving maximum detector performance described in Section 5.5.1 are optional and not required as part of the reverification procedure.

### 5.5.3 Calibration

Prior to sample analysis and after system performance criteria have been satisfied, the instrument is calibrated. All target analytes and all surrogates are present in calibration standards. A list of target analytes and surrogates for volatile and semivolatile analysis appears in Appendix D.3 and D.4, respectively.

#### 5.5.3.1 Initial Calibration

For volatile calibrations five standards at concentrations of 10, 20, 50, 100 and 200 ug/L of all target analytes and system monitoring compounds are prepared. For analysis of volatiles in low level soils (heated purge) a separate calibration is required. For analysis of volatiles in water samples and medium level soils (unheated purges) the same calibration is employed. Separate calibrations are required if the purge volume of water samples is varied.

For semivolatile calibrations standards at five concentrations of 20, 50, 80, 120 and 160 ng/2 ul for all target analytes and system monitoring compounds (except as noted below) are prepared. Semivolatile calibrations apply to all samples, regardless of matrix.

The standards are analyzed and response factors (RF) for each analyte and surrogate at each standard concentration are then calculated based on the instrument response relative to the characteristic ions of the specific compounds and the associated internal standard. Characteristic ions for each analyte and surrogate for volatile and semivolatile analysis appear in Appendix D.3 and D.4, respectively. Internal standard assignments for volatiles and semivolatiles analysis appear in Appendix D.5 and D.6, respectively. The characteristic ions for the internal standards appear in Appendix D.5 and D.6.

RF for each specific compound is calculated using the following relationship:

$$RF = (A_x/A_{is}) (C_{is}/C_x)$$

where:

- A<sub>x</sub> is the area of the characteristic ion for the specific compound;
- A<sub>is</sub> is the area of the characteristic ion for the internal standard used to calculate the concentration of the specific compound;
- C<sub>is</sub> is the concentration of the internal standard (ng/ul)
- C<sub>x</sub> is the concentration of the specific compound (ng/ul)

The mean RF for each specific compound, the standard deviation of that mean and the percent relative standard deviation (%RSD) are calculated for each analyte and surrogate using standard statistical formulate.

Specifications for minimum average RF and maximum %RSD for specific analytes have been established. These specifications appear in Appendix D.4 and D.5 for twenty-four (24) volatile and fifty-two (52) semivolatile analytes and surrogates, respectively.

An initial calibration is valid for volatile analysis if twenty-two (22) or more of the listed analytes and surrogates meet the criteria, provided:

- 1) the compounds which fail have an average RF greater than 0.010 and a %RSD of 40% or less; and
- 2) the twelve (12) target analytes and surrogates not listed also have an average RF greater than 0.010.

An initial calibration is valid for semivolatile analysis if forty-eight (48) or more of the listed analytes and surrogates meet the criteria, provided:

- 1) the compounds which fail have an average RF greater than 0.010 and a %RSD of 40% or less; and
- 2) the twenty (20) target analytes and surrogates not listed also have an average RF greater than 0.010.

After a valid initial calibration is achieved as described in this section, sample or QC solution analysis may proceed provided; 1) there is time remaining in the twelve hour period allowed since the last valid system performance verification, and 2) the initial calibration standard that is the same concentration as the specified continuing calibration standard meets all the continuing calibration acceptance criteria.

#### 5.5.3.2 Continuing Calibration

When twelve hours have elapsed since the last calibration of the instrument, calibration verification is required. Continuing calibration consists of the following steps:

- 1) Verification of instrument performance using BFB or DFTPP; and
- 2) Verification of calibration using a mid-level working standard solution.

The criteria for verification of instrument performance are identical in all respects to those already noted. If these cannot be satisfied by routine adjustments, the instrument is removed from service and appropriate corrective action taken to restore proper function.

For continuing calibration for volatile analysis a 50 ug/l calibration standard is analyzed. For continuing calibration for semivolatile analysis a standard prepared at 50 ng/2ul is analyzed.

Verification of instrument calibration consists of four elements: 1) evaluation of the new RF values with the required minimum values given in Appendix D.7 or D.8; 2) comparison of the new values for RF with the average values calculated for the initial calibration;



3) comparison of internal standard retention times with those observed in the last valid calibration; and 4) comparison of the internal standard areas with those observed in the last valid calibration.

A continuing calibration is valid for volatile analysis if twenty-two (22) or more of the analytes and surrogates listed in Appendix D.7 meet the criteria for minimum RF, provided:

- 1) the compounds which fail have an RF above 0.010;
- 2) the twelve (12) target analytes and surrogates not listed also have an RF greater than 0.010;
- 3) the absolute value of the percent difference between the RF calculated from continuing calibration data and the average RF calculated from initial calibration data is 25% or less;
- 4) retention times observed for internal standards during continuing calibration analysis are within +/- 30 seconds of those observed during the last valid calibration; and
- 5) the area counts for all internal standards are within the limits -50% to 200% of compared to those observed in the last valid calibration.
- 6) % RSD for the twenty-two compounds is greater than 40%.

A continuing calibration is valid for semivolatile analysis if forty-eight (48) or more of the listed analytes and surrogates meet the criteria, provided:

- 1) the compounds which fail have an RF greater than 0.010;
- 2) the twenty (20) target analytes and surrogates not listed also have an RF greater than 0.010;
- 3) the absolute value of the percent difference between the RF calculated from continuing calibration data and the average RF calculated from initial calibration data is 25% or less;
- 4) retention times observed for internal standards during continuing calibration analysis are within +/- 30 seconds of those observed during the last valid calibration, and
- 5) the area counts for all internal standards are within the limits -50% to 200% of compared to those observed in the last valid calibration.
- 6) % RSD for the twenty-two compounds is greater than 40%.

If all of these conditions are met, then calibration has been verified and sample analysis may proceed. If any one of the conditions is unsatisfied, calibration verification has failed. The instrument is removed from service and appropriate corrective action taken to restore proper function.

## 5.6 Gas Chromatography

In general methods in use for gas chromatographic analysis at ARDL are taken from references 1 and 2 cited in the introduction of this document. Procedures for instrument setup, standards preparation and analytical system calibration appear in the paragraphs below. Details relative to sample preparation and analysis are given in Section 6.0. Specifics regarding quality control procedures are given in Section 7.0.

When CLP protocol is required for a sample set, the procedures and requirements given in the most recent USEPA document entitled Statement of Work for Organics Analysis are followed. The procedures detailed in the referenced document are not described herein.

## **5.6.1        Preliminary Preparations**

### **5.6.1.1      Instrument Setup**

Except for volatiles analysis by Methods 601/602 and 8010/8020 for which packed columns are employed, all gas chromatographic analyses are performed using megabore capillary columns (0.53 mm ID) of fused silica. Except when shut down for repair or maintenance, all GC's at ARDL remain in a standby condition, ready to begin the calibration procedures described below.

### **5.6.1.2      Preparation of Standards**

As already noted, all working standards for both initial and continuing calibration procedures are prepared from stock solutions obtained from commercial sources or by weighing appropriate quantities of reagent chemical and diluting to volume with the required solvent. All stock standard solutions are assigned unique code numbers and details relative to their origin and preparation are recorded in a standards logbook as described in Appendix A.1, Standard Operating Procedure, Preparation of Standard Solutions.

### **5.6.1.3      Retention Time Windows**

Retention time windows for the target analytes are determined as described in Section 7.5 of Method 8000, SW-846 whenever a new column is installed or instrument maintenance is performed which will impact on retention time values. Three injections of each analyte are made over the course of a 72 hour period. The standard deviation of the retention time observed for each analyte is determined using standard statistical calculations. For multiresponse analytes a single major peak in the pattern is selected for the calculation.

The retention time window is three times the calculated standard deviation. In those cases where the standard deviation is zero for a particular compound, the standard deviation of the retention times observed for a similar, close eluting compound is utilized.

## **5.6.2        Calibration Procedures**

### **5.6.2.1      Initial Calibration**

With the special exceptions noted below, five point calibration curves are prepared for all analytes of interest and required surrogates. The initial five point calibration for Methods 608/8080, however, includes only the single response pesticides, Arochlor 1254 and the two surrogates, dibutylchloronate and tetrachloro-m-xylene. Single point calibrations for the remaining Arochlors and the multiresponse pesticides are performed as part of this initial

calibration procedure to set retention times and aid in identification of those analytes in the samples to be analyzed.

In cases when a multiresponse analyte is identified during sample analysis, the analytical system is subsequently calibrated at five points for that analyte. The sample(s) in which surrogates, dibutylchloronate and tetrachloro-m-xylene. Single point calibrations for the remaining Aroclors and the multiresponse pesticides are performed as part of this initial calibration procedure to set retention time and aid in identification of those analytes in the samples to be analyzed.

In cases when a multiresponse analyte is identified during sample analysis, the analytical system is subsequently calibrated at five points for that analyte. The sample(s) in which the analyte was identified is then reanalyzed and the concentration of the analyte in the sample determined.

The target analytes and the detection and practical quantitation limits for organochlorine pesticides/PCB's, volatile organics, chlorophenoxy herbicides and organophosphorous pesticides are listed in Tables E.1 through E.4 in the appendix. The analyte list and practical quantitation limits listed in the tables are taken directly from the appropriate methods in SW-846. Detection limits for the organochlorine pesticides, however, were determined experimentally. Detection limits for all other analytes are derived from values given in SW-846.

The low standard is set at a level slightly above CRDL and the high standard is set at the top of the expected linear range for each analyte. A calibration factor is calculated for each analyte at each concentration using the following relationship:

$$CF = (\text{total peak area})/(\text{concentration of standard in ng/ul})$$

For multiresponse analytes total peak area is the sum of the areas of all of the preselected, characteristic peaks appearing in the identifying pattern for the analyte.

Linearity is assumed if the %RSD of the mean calibration factor is less than 20% over the working range of the curve. If the criterion for linearity is satisfied, the mean calibration factor is used for calculation of sample concentrations. If the criterion is not satisfied, sample concentrations are determined using one of two procedures: 1) by regression equation, provided the equation employed has a correlation coefficient of at least 0.995; or 2) from standard curves prepared by plotting instrument response against concentration or mass injected. Best analytical judgement is applied in the latter situations and if, in the opinion of the analyst, neither approach provides suitable data, the instrument is recalibrated and the sample data are reacquired.

#### 5.6.2.2 Calibration Verification

The calibration of the analytical system is verified after analysis of ten samples and at the end of each analytical run. Verification consists of analysis of a mid-range standard containing all of the target analytes. A calibration factor for each analyte is determined as

described above. For analytes which produced linear response initially, calibration is verified if the new response for the analyte is within 15% of the mean value calculated from initial calibration data. If the response for a given analyte was non-linear, calibration is verified if the new calibration factor agrees within 15% of the value calculated for the corresponding standard during initial calibration. If calibration is verified, sample analysis is continued.

If the acceptance criteria are not satisfied the analytical system is recalibrated at five data points as described in the preceding section. The recalibration usually extends to all analytes and surrogates regardless of whether or not they have satisfied calibration verification requirements. On a case by case basis, however, the Quality Assurance Administrator may invoke the standard provisions of Methods 608/8080 and require full recalibration only for the analyte(s) which fail the verification criterion.

## **5.7            Thermometers**

All thermometers in use at ARDL have been calibrated using standard and accepted laboratory procedures against a certified NBS thermometer. Thermometers when received are initially calibrated and recalibrated yearly or as required. This calibration data is recorded on Form QA-7 (see Appendix B.4) and retained permanently in the laboratory files.

### **5.7.1        Daily Temperature Record**

Temperatures in all refrigerated sample storage areas are recorded each working day in a dedicated logbook. Appropriate corrective action is taken when the temperature in one of these areas is not within specifications. A note describing that action is made in the logbook.

The thermometers used for determining these temperatures were calibrated against a certified NBS thermometer as described above. These records are retained permanently in the laboratory files.

## **5.8            Radiation Detection**

Contingent upon prior agreement with specific customers, ARDL may screen incoming samples for radioactivity. Methods employed for that screening are described in Appendix G.

## 6.0 ANALYTICAL PROCEDURES

### 6.1 Inorganics

#### 6.1.1 Methods

Analytical methods in use at ARDL for inorganic analytes are taken from references 1,2 and 3 cited in the introduction of this document. Specific methods employed for each analyte are listed in Appendix G. Details relative to sample preparation and analysis appear in the paragraphs below. Specifics regarding quality control procedures are given in Section 7.0.

When CL methods and protocol are required for a sample set the procedures employed for analysis of inorganics are those described in the most recent USEPA documentation entitled "Statement of Work for Inorganic Analysis", CLP methods and procedures are not described in detail herein except as noted.

#### 6.1.2 Sample Preparation

##### 6.1.2.1 Waters

Before beginning preparative steps the pH of all water samples is to be determined and recorded in the preparation logbook to verify sample preservation. Complete records of the details relative to digestion of each sample set are maintained in the preparation notebook. The records are keyed to a sequential digestion batch number assigned by preparation laboratory personnel. As appropriate, sample sets of similar matrix may be combined in a single digestion batch.

Samples to be analyzed for metals by GFAA, FLAA or ICAP are acid digested using either a hot plate or microwave oven as an energy source. The USEPA CLP procedures are utilized for these digestions. There is a separate procedure for hotplate digestion of analytes to be determined by GFAA (except antimony) and another for analytes to be determined by FLAA or ICAP. A single microwave oven digestion procedure is suitable for all analytical methods. These procedures are summarized below.

For analysis of metals (except antimony) by GFAA, water samples may be digested by hotplate using the following procedure:

1. Add a 1 ml aliquot of 50% nitric acid and 2 ml of 30% hydrogen peroxide to 100 ml. of well-mixed sample.
2. The treated sample is covered and heated to 95C for 2 hours or until sample volume is reduced to 25 to 50 ml. The sample is not allowed to boil during this time.
3. The sample is cooled and insoluble material removed by filtration.
4. Digested sample volume is adjusted to 100 ml with reagent water and analyzed.

For analysis of metals by ICAP or FLAA and for analysis of antimony by GFAA, water samples may be digested by hotplate using the following procedure:

1. Add a 2 ml aliquot of 50% nitric acid and 10 ml of 50% hydrochloric acid to 100 ml. of well-mixed sample.
2. The treated sample is covered and heated to 95C until sample volume is reduced to 25 to 50 ml. The sample is not allowed to boil during this time.
3. The sample is cooled and insoluble material removed by filtration.
4. Digested sample volume is adjusted to 100 ml with reagent water and analyzed.

As an alternative procedure, water sample digestion for all metal analyses may be accomplished using microwave energy. The microwave oven employed meets all specifications given in the primary method reference cited above. Power output of the oven is calibrated regularly according to the procedures cited in that reference.

1. A well-mixed 45 ml aliquot of the sample and 5 ml of concentrated nitric acid are placed in a properly cleaned digestion vessel.
2. The weight of the filled vessel is determined to the nearest 0.02 g and the weight recorded in the digestion log.
3. The vessels are closed with caps fitted with pressure relief valves, inserted into the oven carousel and connected by vent tubing to the cleaned collection vessels already in place. All positions on the carousel are filled either with vessels containing sample material or vessels containing 45 ml of reagent water and 5 ml of concentrated acid.
4. With the samples at an initial temperature of 24° +/- 1°C power is applied for 20 minutes (109 Watts/vessel in the first 10 minutes and 68.8 Watts/vessel in the second 10 minutes).
5. After cooling thoroughly the vessels are uncapped and weighed to the nearest 0.02 g. If a loss in weight exceeding 0.5 g is observed the digestate is discarded and another sample prepared.
6. The digestates are filtered to remove insolubles, brought to a final volume of 50 ml with reagent water and analyzed.

#### 6.1.2.2 Soils, Sludges and Sediments

Samples to be analyzed for any of the target metals by GFAA, FLAA or ICAP are acid digested using either a hot plate or microwave oven as an energy source. These procedures are detailed in Method 3050 of the primary method reference already cited and are summarized below.

##### Hotplate Digestion

1. The sample is thoroughly mixed and 1 to 1.5 g (as is basis) is weighed to the nearest 10 mg.

2. Immediately after taking the sample for digestion, an additional 5 to 10 g is withdrawn for total solids determination using Method 160.3, 40 CFR Part 136.
3. A 10 ml aliquot of 50% nitric acid is mixed with the sample and the preparation is refluxed 95°C for 10-15 minutes without boiling.
4. The sample is removed from the heat and after cooling a 5 ml aliquot of concentrated nitric acid is added. The preparation is refluxed for 30 minutes at 95°C without boiling or allowing the volume of liquid to go below 5 ml. This step is repeated until digestion is complete.
5. When reflux is complete the sample is removed from the heat and allow to cool before adding 2 ml reagent water and 3 ml 30% hydrogen peroxide.
6. The peroxide reaction is initiated by heating and is allowed to proceed with continued heating until effervescence subsides.
7. Peroxide is added in 1 ml aliquots until effervescence is minimal, sample appearance is unchanged or a total of 10 ml has been added.
8. If the sample is being prepared for GFAA analysis of arsenic, beryllium, cadmium, chromium, cobalt, copper, lead, iron, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium or zinc, the procedure in Step 9 is followed. If the sample is being prepared for GFAA, FLAA or ICAP analysis of other analytes, the additional reflux with hydrochloric acid is performed.
9. Heating is continued until the volume of the liquid in the digestate is reduced to approximately 2 ml. A 10 ml aliquot of reagent water is added, insolubles are removed by filtration, and the filtrate brought to a volume of 100 ml with reagent water.
10. The hydrochloric acid reflux required for some analytes involves the addition of 5 ml concentrated acid and 10 ml reagent water to the digestate of Step 7 and refluxing for 15 minutes.
11. When heating is complete, the sample is cooled, filtered and diluted to 100 ml with reagent water and analyzed.

#### Microwave Digestion

1. The sample is thoroughly mixed and 0.5 +/- .050 g (as is basis) is added to the microwave digestion vessel.
2. Immediately after taking the sample for digestion, an additional 5 to 10 g is withdrawn for total solids determination using the procedure referenced above.
3. An aliquot of 10 +/- 0.1 ml of concentrated nitric acid is added to the vessel.
4. The weight of the filled vessel is determined to the nearest 0.01 g and the weight recorded in the digestion log.
5. The vessels are closed with caps fitted with pressure relief valves, inserted into the oven carousel and connected by vent tubing to the cleaned collection vessels already in place.
6. The samples are irradiated for 10 minutes.
7. After cooling thoroughly, the vessels are uncapped and weighed to the nearest 0.01 g. If a loss in weight exceeding 10% of the original weight is observed the digestate is discarded and another sample prepared.

8. The digestates are filtered and brought to a final volume of 50 ml. with reagent water and analyzed.

### 6.1.3 Sample Analysis

Analytical runs are planned by preparation of a preliminary run log. The log indicates: 1) the calibration standards to be analyzed; 2) applicable initial quality control samples to be evaluated; 3) the samples for analysis; and 4) applicable calibration verification standards and quality control data points.

Using this log as a reference, the run is initiated. Once an acceptable calibration has been achieved and the applicable initial quality control requirements have been satisfied, sample analysis begins in accordance with log entries.

Unless there is specific reason to suspect a high concentration of an analyte, all sample digestates are initially analyzed undiluted. When an analyte concentration exceeds the linear range for ICAP analyses or the calibration range for other analyses, the digestate is diluted. Based on instrument readings obtained on the undiluted digest, the dilution required to bring analyte concentration within the linear or calibrated range is estimated and the digestate is reanalyzed.

For all analyses by GFAA a minimum of two separate injections of the sample digestate is required. The individual measurements as well as their mean and relative standard deviation are generated by the instrument data system. If the measured analyte concentration exceeds CRDL, the RSD of the individual measurements must be equal to or less than 20%. If this criterion is violated, the determination is repeated once. If the RSD criterion is violated in both analyses, the sample result with the lowest RSD is reported.

ICAP measurements require a minimum of two replicate exposures during aspiration of the sample. There are no limitations on the precision which must be achieved by these replicates.

Sample analysis proceeds as long as the applicable quality control and calibration verification requirements are satisfied. Acquisition of quality control and calibration verification data points is required based on the number of analytical samples evaluated and/or time. Specifications relative to calibration verification appear in Section 5, above. The measurements defined as analytical samples and the frequencies required for acquiring quality control data appear in Section 7.1.1, below.

If one of these measurements falls outside of the acceptable window, corrective actions are taken as appropriate. As described in the sections of this document referenced above, the extent of the corrective action and the steps taken to verify the effectiveness of that action is dependent on the problem encountered.



#### 6.1.4 Calculation of Sample Concentrations

Observed transmittance or absorbance on samples and QC solutions are recorded in the analysts logbook for instruments without an associated data system. In all cases the calculations utilized for determination of concentration values based on these readings are clearly shown. Details relative to analyst logbook records appear in Appendix A.2, Standard Operating Procedure, Instrument Records and Logbooks.

For instruments with integral data systems the observed values for absorbance or transmittance are printed as the data are acquired. At the discretion of the analyst, some data systems will calculate sample concentrations directly. In such cases, appropriate sample weights or volumes, dilutions and other correction factors are made known to the data system by keyboard entry. Printed values are time/date stamped by the data system. The originals of these raw data are retained on file indefinitely in the laboratory.

For instruments without integral data systems, sample concentrations are calculated by hand and the values obtained are recorded in the analyst's logbook directly or transferred onto preprinted analytical run report forms. Reduced photocopies of these forms are subsequently affixed to pages in an analytical logbook containing other data acquired on the same instrument. Logbooks are retained on file indefinitely in the laboratory.

Calculation of sample concentrations in soils is performed as follows:

$$\text{mg analyte/Kg dry sample} = [(C \times V)/(W \times S)] * \text{Dilution Factor}$$

where: C = digestate concentration in mg/L (instrument reading)  
V = final volume in liters after sample preparation  
W = wet sample weight taken expressed in kg.  
S = 0.01 x %solids

#### 6.2 Organics

##### 6.2.1 Methods

Except for analyses involving gas chromatography/mass spectrometry, the analytical methods in use at ARDL for organic analytes are taken from either of three references: 1) "Test Methods for Evaluation Solid Waste: Physical/Chemical Methods", SW-846, 3rd Ed., USEPA, November 1986; 2) 40 CFR Part 136, Federal Register, October 26, 1984; or 3) "Standard Methods for the Examination of Water and Wastewater", 16th Ed., 1985. Calibration and QC requirements relative to GC/MS analyses by Methods 8240, 8260, 8270, 8310, 624 and 625 are taken from USEPA Document OLM01 (all revisions up to and including OLM01.7 dated July, 1991), entitled Statement of Work for Organics Analysis. The specific methods used for determination of organic analytes are listed in Appendix G.

## 6.2.2 Sample Preparation

### 6.2.2.1 Purge and Trap

The purge and trap procedures described in Method 5030 (SW-846) and Method 624 (CFR 136) are used for preparation of samples for determination of volatile components. For these determinations, sample preparation and sample introduction to the analytical system are integrated into one procedure. That procedure is summarized below.

A measured quantity of sample is introduced into the purging vessel. The necessary surrogates and internal standards are added directly to water and water miscible samples. Soil and other solid samples are suspended in an appropriate volume of water which contains the required surrogates and internal standards.

The purging vessel is connected to the purge and trap device and purged with inert gas as specified by the subject methods. Purging vessels containing soil and other solid samples are heated to 40°C. The stream of purging gas is directed to a sorbent trap which collects the volatile components in the sample. When the purging interval has elapsed, the trap is rapidly heated to 180°C while being backflushed with inert gas. The backflush stream is directed to an appropriately calibrated analytical instrument.

### 6.2.2.2 Extractions

#### 6.2.2.2.1 Water

Water samples are prepared for analysis using either Method 3510 (Separatory Funnel Liquid-Liquid Extraction), modified to conform to procedures given in the USEPA document referenced above. These procedures are summarized below.

#### Separatory Funnel

1. A 1.0 L aliquot of sample and the required aliquots of surrogate and spiking solutions are transferred to a 2 L separatory funnel.
2. Sample pH is adjusted appropriately for extraction of the desired analytes.
3. The sample is extracted by shaking for 1-2 minutes with three separate 60 ml portions of methylene chloride. The extracts are combined.
4. If a secondary extraction is required to obtain a second fraction from the sample, Steps 2 and 3 are repeated.
5. The combined extracts are dried by passing through a 20 mm diameter drying column filled with approximately 10 cm of anhydrous sodium sulfate. The dried extract is collected in a Kuderna-Danish concentrator assembly.
6. The vessel which had contained the combined extracts and the drying column are rinsed with 20-30 ml of methylene chloride and the rinsate is collected in the concentrator assembly with the dried extract.

7. The dried extract is concentrated to a final volume of 1 ml. The concentrated extract is ready for cleanup, solvent exchange or analysis, dependent on the target analytes.

#### **Liquid-Liquid Extractor**

1. After closing the sidearm stopcock on the liquid-liquid extractor (Corning One-Step, Catalog No. 2550-xx with continuous drier adapter), approximately 400 ml of methylene chloride is placed in the assembly.
2. The pH of 1.0 L of sample is adjusted appropriately for extraction of the target analytes and added to the extractor assembly.
3. The required surrogate and spiking solutions are added.
4. The flow of cooling water (4°C) to the reflux condensers and of hot water (80°C) to the concentrator is started.
5. The sidearm stopcock is opened to equilibrate liquid levels and to initiate operation of the apparatus.
6. The sample is extracted for 18-24 hours.
7. When extraction is complete, the sidearm stopcock is closed and the volume of methylene chloride in the concentrator is reduced to 1 ml. The concentrated extract is ready for cleanup, solvent exchange or analysis, dependent on the target analytes.
8. The methylene chloride distilled from the concentrator is directed to the main body of the extraction unit where it combines with the extracted sample. This waste is retained for later disposal.

#### **6.2.2.2.2     Soil**

Soil samples are extracted using Method 3550 (Sonication Extraction). The procedures is summarized below.

1. Free standing water and foreign object such as stones, sticks, leaves, etc., are discarded and the sample is mixed well.
2. Two sample aliquots are weighed; one for subsequent extraction and one for determination of moisture using Method 160.3.
3. The sample to be extracted is placed in a 400 ml beaker and mixed thoroughly with 60 g of anhydrous sodium sulfate.
4. Surrogate and spike solutions are added as required.
5. A 100 ml aliquot of 1:1 methylene chloride:acetone is added and the preparation is sonicated for 3 minutes.
6. The extracting solvent is decanted through filter paper and the sonication is repeated twice more using additional 100 ml aliquots of solvent.
7. On completion of the final sonication, the entire sample is added to the filter apparatus. The sample and the sonication vessel are washed with additional amounts of extracting solvent.

8. The combined extracts are dried by passage through 20 mm diameter drying column filled with 10 cm of anhydrous sodium sulfate and collecting the dried extract in a Kuderna-Danish concentrator.
9. The column and the vessel which contained the wet extract are washed with additional quantities of solvent which are collected in the concentrator.
10. The extract is concentrated to a final volume of 1 ml. The concentrated extract is ready for cleanup, solvent exchange or analysis, dependent on the target analytes.

#### **6.2.2.2.3      Extract Cleanup**

As required, sample extracts are subjected to appropriate cleanup procedures. Facilities for sample cleanup using Methods 3640 (Gel Permeation Cleanup), 3620 (florosil Column Cleanup), 3610 (Alumna Column Cleanup) and 3660 (Sulfur Cleanup) are in place in the laboratory. When any of those procedures are to be employed, the adjustments are made in levels of surrogate/spike and final extract concentration volumes as required by the methods in use.

#### **6.2.3            Analyte Identification**

##### **6.2.3.1        Retention Time**

Retention time windows are established for all analytes of interest in gas chromatographic analyses. For Methods 8240, 8260 and 8270 the windows are based on the retention times observed for the analytes during calibration. For all other gas chromatographic analyses, windows are established using the procedures described in Section 5.6, above. Instrument conditions employed for establishing those windows are identical to those employed during sample analysis.

These windows are used as a means of identifying target analytes in all gas chromatographic analyses. For analyses in which mass spectral data are collected on the analytes, retention time is one of two criteria employed. For all other analyses, retention time is the only criterion used for analyte identification.

If a peak is observed within a window for a target analyte during sample analysis by gas chromatography, the first presumption is that the peak reflects the presence of the analyte in the sample. For analyses in which only retention time is available as a means of analyte identification, the detect is confirmed by analysis on another instrument with a column of differing properties. For analyses in which mass spectral data are obtained the identity of the sample is confirmed by evaluation of those data as described in the following paragraph.

##### **6.2.3.2        Mass Spectra Interpretation**

The NBS Mass Spectral Library and standard spectra generated with ARDL's equipment are available on line for use in data system library searches for identification of sample components. For confirming the identity of sample components using mass spectral data, ARDL analysts observe the following general guidelines.

- Ions present in the standard mass spectrum at more than 10% relative intensity should be present in the sample spectrum;
- The relative intensities of those ions in the sample should agree within +/- 20% of the relative intensities in the standard spectrum;
- Ions present in the library reference spectrum should be present in the sample spectrum;
- If some ions in the reference spectrum are missing from the sample spectrum, the data should be reviewed to confirm the ions have not been subtracted from the sample by the software in order to compensate for background or coeluting peaks.

In addition to adhering to these guidelines, analysts are encouraged to rely on their best judgement in evaluation of mass spectral data for purpose of sample component identifications.

#### 6.2.4 Sample Analysis

All analytical systems employed are initially calibrated as described in Section 5, above. Sample analysis proceeds uninterrupted as long as the calibration verification specifications for the methods employed are satisfied. As required by these methods, appropriate QC samples are analyzed with the samples as described in Section 7, below. Appropriate corrective actions are taken whenever a QC data point falls outside permissible limits.

Except for determination of volatiles by Methods 8240, 624, 601/602 and 8010/8020 for which packed columns are employed, fused silica capillary columns are utilized for all gas chromatographic analyses. A conventional capillary column (0.32 mm i.d.) is used for Method 8270 analyses. All other analyses employ wide bore capillary columns (0.5 mm i.d. and larger). Initial column temperatures, ramps and other specific instrument conditions employed are method dependent.

#### 6.2.5 Calculation of Sample Concentrations

The procedures utilized for calculation of sample concentrations are dependent on sample matrix and whether internal or external standards are employed in calibration of the analytical system. Most sample results are calculated by computer software. Data for the calculations are obtained either by direct reading of analytical instrument output or keyboard input of instrument readings by analysts.

For water samples analyzed using internal standard calibration methods the general equation for calculating analyte concentrations is:

$$\mu\text{g/L} = (A_x * C_{is} * D) / (A_{is} * RF * V_s)$$

Where:

- $A_x$  = analyte response in peak height or area;
- $C_{is}$  = ng of internal standard added to the extract or volume purged;
- $D$  = dilution factor (value of 1 if none were made);

$A_{is}$  = internal standard response using the same units as  $A_x$ ;  
 RF = response factor for the analyte; and  
 $V_s$  = ml of sample purged.

The response factor referenced above is calculated based on instrument readings observed during the most current calibration using the relationship:

$$RF = (A_s * C_{is}) / (A_{is} * C_s)$$

where:  $A_s$  = instrument response for the analyte;  
 $A_{is}$  = instrument response for the internal standard;  
 $C_{is}$  = internal standard concentration in ug/L; and  
 $C_s$  = analyte concentration in ug/L

For soil, waste and solid samples the calculation is identical except that sample weight (in g corrected for moisture) is substituted for sample volume purged ( $V_o$ ). The units for sample concentration are ug/kg.

Calculations for determinations based on external standard calibrations are similar. For waters the general equation is:

$$\text{ug/L} = (A_x * V_t * D) / (CF * V_i * V)$$

$A_x$  = response for the analyte;  
 $V_t$  = total extract volume in uL;  
 $D$  = dilution factor (1 if no dilutions are made);  
 $Cf$  = calibration factor for the analyte based on the current calibration data;  
 $V_i$  = volume of the extract injected in uL (1 for purge and trap); and  
 $V$  = volume of the sample purged or extracted in ml.

The calibration factor (CF) referenced above is calculated from calibration data using the following relationship:

$$CF = \text{Total response by analyte/concentration of standard in ng/ul}$$

For soil, waste and solid samples the calculation is identical except that sample weight (in g corrected for moisture) is substituted for sample volume purge ( $V_o$ ). The units for sample concentration are ug/kg.

## 7.0 QUALITY CONTROL

### 7.1 Inorganic Analyses

#### 7.1.1 Frequency Requirements

The requirements for frequency of quality control checks and calibration verifications are based on the number of analytical samples evaluated. As defined in this document, a frequency of 10% means once every 10 analytical samples. A frequency of 5% means once every 20 analytical samples. Analytical samples are defined herein include customer sample materials as well as some quality control solutions. For purposes of this document, analytical samples include the following:

- undiluted or diluted samples from any source
- predigestion spike samples
- duplicate samples
- serial dilution samples
- analytical spike samples
- post-digestion spike samples
- interference check samples
- laboratory control samples
- preparation blanks
- linear range samples

Frequency requirements for quality control data are as follows:

Preparation Blanks - one for each sample batch or every 20 samples, whichever is more frequent.

Matrix Spikes/Spike Duplicates - one for each matrix (water, soil) in a sample delivery group.

Analytical Spikes - as required by matrix spike results for all analyses.

Duplicates - once for each matrix in a sample batch or once for every 10 samples of the same matrix in a batch, whichever is more frequent.

Laboratory Control Samples - once for every sample digestion batch.

#### 7.1.2 Preparation Blanks

The absolute value of the analyte(s) of interest observed in the preparation blank may not exceed CRDL. When this criterion is satisfied, all sample concentrations observed are reportable, provided all other quality control and calibration verification data requirements are satisfied.

When blank values exceed acceptable limits the sample preparation steps must be repeated and the new extracts or digests reanalyzed. There are, however, two exceptions made to

this general rule. If sample concentrations are 10X or greater than the concentration found in the blank or if sample concentrations are less than CRDL, the sample results are valid and reportable.

### 7.1.3 Matrix Spikes and Spike Duplicates

For each matrix (soil, water) in a sample set a matrix spike and matrix spike duplicate are required. Matrix spike material is added to the sample taken for analysis before beginning any preparation steps.

Matrix spike levels vary according to analyte, analytical method and matrix type. Appendix F.3 lists the required levels.

Matrix spike levels shown for metals in water matrix indicate the concentration of the spike required in the finished digestate. For water samples digested by hot plate, the final volume of digestate is 100 ml. If digested with microwave energy, the final volume is 50 ml. To obtain the specified concentration of antimony (500 ug/l) in the spiked sample, for instance, a total of 50 ug (500 ug x 1.0 liters) of the element is required. To obtain this total, 1.0 ml of a 50 mg/l spiking solution of antimony is pipetted into the sample aliquot taken before digestion begins. If the sample is digested with microwave energy, 0.5 ml of the spiking solution is added to achieve the same result.

Matrix spike levels shown in Appendix F.3 for metals in soil samples indicate the concentration required in the digestate when the relationships of final volume and sample weight (as is basis) for the various analytes are as indicated below:

<u>Analyte</u>	<u>Digestion Procedure</u>					
	<u>Mercury Method</u>		<u>Hot Plate</u>		<u>Microwave</u>	
	<u>g Sample</u>	<u>ml</u>	<u>g Sample</u>	<u>ml</u>	<u>g Sample</u>	<u>ml</u>
Mercury	0.20 - 0.35	100	-----	-	-----	-
Other Metals	-----	-	0.9 - 1.5	100	0.4 - 0.6	100

The calculation for determining the final concentration of the required spike in the digestate is identical to the example given above for water samples. If the sample weight employed or digestate final volume are outside of the tabulated values, a proportional adjustment in the spike amount is required.

Cyanide matrix spike levels in waters are referenced to the volume taken for analysis. The spiking level is 100 ug for each liter analyzed. If only 500 ml are analyzed, 50 ug is added as a spike. If 50 ml are analyzed, 5 ug is added as a spike. The spike is added directly to the sample before distillation begins.

For soil samples, the cyanide matrix spike level is dependent on both the sample weight taken and the final distillate volume. For each gram of sample (as is basis) the final distillate must contain 100 ug/l cyanide spike. If a final distillate of 250 ml is to be obtained from a 1 g sample, the spike amount added before distillation is 25 ug.



Recovery for all spikes is calculated using the following relationship:

$$\% \text{ Recovery} = (100) (\text{SSR}-\text{SR})/\text{SA}$$

where:

SSR is the spike sample result.

SR is the sample result.

SA is the spike amount added.

When the sample concentration is less than IDL a value of zero is used for SR when calculating percent spike recovery. Acceptable limits for matrix spike recovery are 75% to 125%. If sample concentration exceeds the spike amount by a factor of 4X, these limits are waived.

Evaluation of matrix spike and matrix spike duplicate results is summarized in the text below and presented graphically in the decision tree appearing in Appendix F.3. As a first step in that evaluation, results obtained on the matrix spike and the matrix spike duplicate are compared. If recovery on each is within the limits cited above, there is no requirement set for the relative percent difference (RPD) between the two values. If recovery in one of the spikes is outside the range 75 - 125%, both samples are reanalyzed once to confirm the values obtained. If, on reanalysis, both spikes satisfy the acceptance limits, no further action is taken and sample analysis proceeds uninterrupted.

If the initial values of recovery are confirmed, however, a comparison between the spike and spike duplicate results is made. If the RPD is less than 20%, no further action is taken and sample analysis proceeds uninterrupted. If the RPD exceeds 20%, one of two actions is taken, dependent on the analyst's judgment in the matter. Either the spike and spike duplicate are redigested and reanalyzed to confirm the initial observations or all of the associated samples and a new spike and spike duplicate are prepared. In those cases where just the spike and spike duplicate are redigested and reanalyzed, the second set of spike and spike duplicate values are compared. If the results are similar to those observed initially, no further action is taken and sample analysis proceeds uninterrupted. If the results are not comparable, all samples in the set are redigested and reanalyzed.

If recovery in both the spike and the spike duplicate is outside the range 75% - 125%, a comparison of the results observed is made. If the RPD is greater than 20%, sample preparative steps for the entire sample set is repeated and the new digestates reanalyzed. If the RPD is less than 20%, however, the sample is bench spiked and analyzed. Bench spike acceptance criteria and the actions taken based on results obtained are described in Section 7.1.4, below.

If a second digestion and analysis of the samples set culminates in a leg of the decision tree which requires still another redigestion and reanalysis, the customer is advised of the probable matrix interference and of the need to analyze the samples by the method of standard additions (see Section 7.1.4.1, below).

#### 7.1.4 Analytical (Bench) Spikes

Except for the elements mercury and silver, analytical spikes are prepared and analyzed whenever the acceptable limits for matrix spike recovery are violated. Analytical spikes are prepared by adding the appropriate level of the affected element(s) to the digested sample and analyzing the spike sample. As a rule, analytical spiking levels are the same as those used for matrix spikes. Analysts are at liberty, however, to change those levels as appropriate based on sample concentration, instrument calibration range and other constraints. In any case, the spike volume added is limited to 10% or less of the volume of sample digestate which is taken for spiking.

If recovery of the bench spike is acceptable (75% to 125%), sample preparative steps for the entire sample set is repeated and the new digestates reanalyzed. If the recovery on the bench spike is not acceptable, the customer is advised of the probable matrix interference and of the need to analyze the samples by the method of standard additions as described in the section immediately below.

##### 7.1.4.1 Method of Standard Additions (MSA)

MSA consists of adding systematic increments of the analyte of interest to the sample. Assuming zero concentration in the sample, the regression equation for the instrument readings observed is calculated. Sample concentration is then reported as the absolute value or the x intercept, provided the plot is linear. Linearity is assumed when the correlation coefficient,  $r$ , is 0.995 or more.

The following procedures are followed in performance of MSA analyses.

- A. The sample and three spikes are analyzed consecutively for MSA quantitation using single injections.
- B. MSA spikes are selected based on approximate percentages of the estimated sample concentration:

- Spike 1 - about 50% of the sample concentration.
  - Spike 2 - about 100% of the sample concentration.
  - Spike 3 - about 150% of the sample concentration.

- C. Except as noted below, if the level of analyte observed in any of the MSA spikes exceeds the upper limit of the calibration range, the sample is diluted and MSA repeated using spikes of appropriate absorbances. If, however, the correlation coefficient,  $r$ , of the curve obtained is 0.995 or better, no dilutions are made since the value of  $r$  demonstrates that the curve is linear.

- D. If the correlation coefficient,  $r$ , is less than 0.995, the analysis is repeated. If the value of  $r$  is less than 0.995 the second time, MSA is terminated and the result obtained with the highest value for  $r$  is reported.

#### 7.1.5 Duplicates

Duplicate analysis of samples is performed when: 1) there is no suitable spiking compound available; 2) the properties of the analyte (e.g. pH, specific conductance, flash point, etc.) are such that spiking is impractical; or 3) no spikes are required by the method for the analyte (e.g., calcium, magnesium, sodium, etc. See Appendix F.2). For trace metal analytes for which no spikes are required, duplicate determinations are performed at the frequency specified for matrix spikes and spike duplicates. For all other analytes, duplicates are analyzed every ten samples. The relative percent difference (RPD) between the initial analysis and the duplicate is calculated using the following relationship:

$$RPD = (100) (S-D) (1/AVE)$$

where: S is the result of the initial analysis;  
D is the result of the duplicate analysis;  
S-D is the absolute value of the difference; and  
AVE is the mean of S and D.

If the value of both analyses are equal to or greater than five times CRDL, the control limit for RPD is 20%. If the value of either analysis is less than five times CRDL but greater than IDL, the control limit is the CRDL. If either value is less than IDL, no value for RPD is reported.

#### 7.1.6 Laboratory Control Samples

LCS's are analytical standards subject to all preparative steps and analytical procedures used on sample materials received from the field. An LCS is not required for analysis of water samples for mercury or cyanide. Aqueous standards are used as LCS for all samples, regardless of matrix. One LCS is prepared and analyzed for each digestion batch or every twenty samples, whichever is less.

The LCS is intended to gauge the effectiveness of the digestion procedures utilized in sample preparation. As a matter of routine the LCS is analyzed in the same sequence as the samples prepared with it. In some situations, however, it may not be possible to acquire all of the sample data in that run. In these cases, the remaining samples are analyzed at a later time. There is no requirement to reanalyze the LCS once a valid analysis for it has been acquired unless some of the samples are analyzed on another kind of instrument (e.g., samples analyzed by ICAP are also analyzed by flame atomic absorption). In such cases, the LCS is also analyzed on that second instrument.

Percent recovery of LCS is calculated using the following relationship:

$$\%R = 100 (\text{Observed Value}/\text{True Value})$$

where: Observed Value is the result obtained on analysis of the LCS; and  
True Value is the known concentration of the analyte in the LCS.

Acceptable percent recovery for all analytes in aqueous LCS is 80-120% of true value. Antimony and silver are excepted from that limitation.

If results of the LCS fall outside the acceptable limits, analysis is suspended and corrective action is taken. At the option of the analyst, immediate reanalysis of the LCS is permitted as a first corrective action to confirm that a problem has been encountered. If reanalysis shows that the first evaluation was in error, continuing calibration verification is performed as described in Sections 5.3.2 or 5.4.2, above, to reaffirm proper function of the analytical system. If calibration is verified, sample analysis is resumed.

If the optional first corrective action shows that a problem exists or the analyst elects to bypass that step, other corrective actions are taken as appropriate. When the problem has been identified and the analytical system is restored to proper function, it is recalibrated and sample analysis reinitiated after acceptable results are obtained on the LCS in question. If it is determined that the poor recovery of the LCS is associated with an inadequate digestion, all samples associated with the LCS which failed are redigested and reanalyzed.

#### 7.1.7 Serial Dilution Analysis (ICAP)

When an unusual sample matrix is encountered, serial dilution analysis of a sample from that matrix is performed prior to reporting sample concentrations. The sample is diluted 5X with reagent water and analyzed. The values observed in the diluted sample are corrected for that dilution. The corrected values and those obtained upon the initial valid analysis of the sample digestate are compared. Percent difference is calculated as follows:

$$\%D = (100) (I-S)/I$$

where: I is the initial result on the sample digestate;  
S is the serial dilution result.  
I-S is the absolute value of the difference observed.

If the values observed on both the initial and serial dilution analyses are below IDL, percent difference is not calculated. If the uncorrected value observed in the serial dilution analysis is below IDL, a zero is used for S in calculation of percent difference.

For those analytes at concentrations of less than 50X IDL in the original sample serial dilution results have no significance. For those analytes at concentrations of 50X IDL and higher in the original sample, however, the percent difference between the initial and serial dilution analysis should be no more than 10%. If this value is exceeded the presence of a chemical or physical interference is suggested. The customer is notified of the problem and, upon his authorization, further evaluation of the samples is performed.

## **7.2            Organics**

### **7.2.1        Frequency Requirements**

The requirements for frequency of quality control checks and calibration verification are based on the number of analytical samples evaluated. As defined in this document, a frequency of 10% means once every 10 analytical samples. Analytical samples as defined herein include customer sample materials as well as some quality control solutions. For purposes of this document, analytical samples include the following;

- undiluted or diluted samples;
- duplicates;
- matrix spikes; and
- preparation blanks

Frequency requirements for quality control data are as follows:

Preparation Blanks - one for each sample batch or every 20 samples, whichever is more frequent.

Matrix Spikes - one for each matrix (water, soil) in a sample delivery group.

Duplicates - once for each matrix in a sample batch or once for every 20 samples of the same matrix in a batch, whichever is more frequent.

### **7.2.2        Demonstration of Precision and Accuracy**

For all methods listed in Appendix G.2 ARDL has historical data to demonstrate that the levels of precision and accuracy achieved in the laboratory are comparable to those required by the methods.

### **7.2.3        Blanks**

A procedural blank is analyzed for each batch of samples of similar matrix prepared in the laboratory. Procedural blanks are reagent water or reagent grade sodium sulfate carried through all steps necessary for preparation of samples for analysis.

Except as noted below, procedural blanks may not contain levels of target analytes greater than the CRDL values given in Appendix D and Appendix E. The common phthalate esters may be present in blanks at 5X CRDL. For blanks prepared with samples intended for volatile analysis, the common laboratory solvents methylene chloride, acetone and 2-butanone may be present at 5X CRDL.

Analysis of samples may not proceed until the procedural blank associated with their preparation satisfies the criteria above. If the procedural blank does not satisfy these requirements the sample preparation steps are repeated.

#### Surrogates

Prior to beginning sample preparation, surrogate compounds are added to all samples, blanks and laboratory controls to evaluate the effectiveness of the preparative steps. The identity of the surrogates varies, dependent on method. The specific compounds used as surrogates and the levels at which they are added to the samples are tabulated in Appendix D and Appendix E. Where no surrogate is identified, a target analyte not expected in the samples is employed as one. In such cases it is usual to add the surrogate to the sample at a level which, in the final extract, will be approximately 75% of the calibration range employed for the analysis.

Surrogate recovery is expressed as percent and is calculated by dividing the concentration observed in the sample by the true value and multiplying the results by 100. Limits for surrogate recovery are fixed by the protocol used for Methods 8240, 8260 and 8270. These limits are tabulated in Appendix D.

Advisory limits for surrogate recovery for other methods are established using the procedures outlined in Method 8000, Section 8.10. Percent surrogate recovery is calculated for at least thirty samples of each sample matrix (soil or water) analyzed. The mean and standard deviation of the values obtained is calculated. The high limit for surrogate recovery is equal to the mean of observed recoveries plus three times the standard deviation. The low limit is the mean less three times the standard deviation. Limits for surrogate recovery are reevaluated on a semiannual basis.

As indicated, surrogate recoveries are advisory in all cases except where limits are specified by method. In those cases where there are method specifications, if surrogate recovery falls outside control limits in a procedural blank, sample preparation is repeated for all samples associated with that blank. If surrogate recovery falls outside control limits in a sample, the sample is reanalyzed. If surrogate recovery is within limits on reanalysis, only the results of the reanalysis are reported. If surrogate recovery is still outside control limits, the results of both analyses are reported and the customer is notified of the probable sample matrix interference.

#### Matrix Spikes

Every twenty samples of similar matrix or once each sample set, whichever is more frequent, a sample is spiked in duplicate with a specific compound or battery of compounds at specified concentrations. The identity of the spiking compounds and the concentrations specified are method dependent. Spiking requirements are tabulated in Appendix D and Appendix E. If no spike requirements are specified by the method employed, an unspiked sample is analyzed in duplicate at the frequency cited above.

Spike recovery is expressed as a percent and is calculated using the relationship given below.

$$\% \text{Recovery} = (100) (\text{SSR} - \text{SR}) / \text{SA}$$

where: SSR is the spike sample result.  
SR is the sample result.  
SA is the spike amount added.

Advisory control limits for spike compound recovery for each kind of sample matrix evaluated are determined using the same procedures outlined above for surrogate recovery control limits. Percent spike recovery is calculated for at least thirty samples of deviation of the values obtained is calculated. The high limit for spike recovery is equal to the mean of observed recoveries plus three times the standard deviation. The low limit is equal to three times the standard deviation subtracted from the mean. Limits for spike recovery are reevaluated on a semiannual basis.

No corrective action is required if spike recovery is outside the control limits.

### 7.3 Detection Limits

#### 7.3.1 Inorganics

Instrument detection limits (IDL) or method detection limits (MDL) for inorganic target compounds are determined experimentally. IDL is a measurement of the signal to noise ratio of a specific analytical system performing under a given set of conditions. MDL is the minimum concentration of an analyte which can be measured using a specific analytical method and reported with 99% confidence that the value is above zero.

Detection limits for target metal analytes and cyanide are determined on a quarterly basis. Detection limits for other analytes are determined on an approximately annual basis. Detection limits are estimated based on previous history, knowledge of the sensitivity of the test involved or the recommendations of the instrument manufacturer. Standard solutions of the target analytes are prepared separately in reagent water at concentrations of 3-5 times this estimated concentration.

For trace metal analytes, the concentration in these solutions is determined on three non-consecutive days with seven consecutive measurements per day. The standard deviation of the values obtained on each day is calculated. The IDL is the sum of those standard deviations. An IDL for metals is usually reported in ug/L.

For other inorganic analytes, the concentration in these solutions are measured at least ten times. The measurements are more or less evenly divided between at least two analytical sequences. The IDL (or MDL) is equal to three times the standard deviation of the measurements made. The detection limit is reported in terms of ug/L or mg/L, dependent on the sensitivity of the analysis involved.

Raw data from IDL determinations are retained on file indefinitely in the laboratory.

MDL's for selected organic analytes are determined on an approximately annual basis using the procedures described by Glaser et al in Analytical Chemistry, Volume 15, Number 12, December, 1981. These procedures involve at least seven consecutive analyses of standards of a known concentration that is approximately 2 to 5 times the estimated MDL. The MDL is the standard deviation of the results obtained multiplied by Student's "t" value (99% confidence level, n-1 degrees of freedom). The MDL's determined in this manner are essentially equivalent to the values given in the methods listed in Appendix G.



## **8.0            DATA HANDLING AND REPORTING**

### **8.1            Laboratory Data System**

ARDL employs a PC based laboratory information management system (LIMS) for handling data in the laboratory. The system (Smartlab, Telecations, Inc.) is developed on Smartware (Informix Software, Inc.), a well-known relational database with spreadsheet and word processing capabilities. The LIMS is networked and various nodes are located appropriately throughout the laboratory. Password security for the various users permits access to the portions of the database required for performance of their duties.

As indicated previously, all samples submitted to ARDL are logged-in to the LIMS. In accordance with good automated laboratory practice, each sample is assigned a unique number by which it is known to the LIMS. Provisions have been made for generation of a second number which relates all samples in a given sample group. This second number is ergonomically more appropriate and is utilized by laboratory personnel in every day references to the sample.

Provisions also have been made in the database for automatic calculation of: 1) dates for completion of analytical work based on promised turnaround; 2) sample holding times as a function of customer requirements and sample matrix; and 3) automatic calculation of critical dates relative to sample preparation and analysis. Quality control samples are added to the database automatically based on the requirements outlined in Section 7, above, or on customer specific requirements which have been agreed upon prior to sample receipt.

Analytical group and section supervisors use the system for assignment of analytical work. The system functions largely on the basis of analytical runs and both analysts and supervisors design these runs based on sample load, critical dates and other considerations. Among the many capabilities of the system are provisions for; 1) display of calibration curves, 2) calculation of sample results, 3) supervisor evaluation of QC sample results, and 4) comparison of current and previous data from a given customer.

### **8.2            Recording Analytical Data**

Permanent records and logs are generated for all operations in the laboratory involving sample preparation and sample analysis. Analytical data and sample preparation information generated in the laboratory are in one of three formats; 1) instrument output in hard copy and/or computer data files, 2) handwritten transcriptions of readings taken from instrumentation, or 3) handwritten descriptions of laboratory activities. All analytical data except that generated by the GC/MS data system are entered into the LIMS. Transfer of the data into the LIMS is accomplished by keyboard entry or electronic transfer, either directly from an instrument or by diskette.

#### **8.2.1        Laboratory Logbooks**

When it is necessary to prepare handwritten notations of procedures followed or records of instrument readouts, those data are transcribed directly into bound logbooks or, in

some cases, on preprinted forms which are subsequently affixed to pages in such logbooks. The procedures followed in maintenance of these logbooks are described in detail in Appendix A.1 through A.3. Once the appropriate logbook entries have been completed, results for field and QC samples are entered into the LIMS by keyboard either by the analyst or the LIMS operator.

#### **8.2.2      Instrument Output (Data Files and Hardcopy)**

Instrument output in either tabular or graphic form is retained on file indefinitely in the laboratory. In those cases where the instrument data system generates a computer data file, that file rather than the hard copy output may be the format in which the raw data records are retained.

In general computer files generated by an instrumental data system will save those files on a high capacity hard drive. Internal procedures are in place for transfer of these files to diskettes to guard against data loss because of hard drive failure and to keep an adequate portion of the drive available for new data acquisition. These backup procedures are carried out on a weekly basis as a minimum and more frequently when sample throughput on a given instrument system is high.

#### **8.3      Verifying Analytical Data**

Analysts are responsible for ensuring that data which they have acquired meets all applicable specifications relative to calibration and quality control outlined in Sections 5 and 7 of this document. Further, they are responsible for ensuring that the sample concentrations which they report have been accurately calculated.

Once the analyst has completed work on a sequence or with a group of samples, the raw data and all calculations are subject to peer review for accuracy and completeness and adherence to the method employed. When peer review is complete sample results and QC values are input the LIMS, either by keyboard entry or by electronic transfer directly from the instrument or by diskette.

The raw data and reported values are subsequently evaluated by the section or group supervisor. That review is made on screen by consulting data in the LIMS and, where appropriate, by review of logbooks or raw data printouts. It consists of a spot check for accuracy in calculations, evaluation of the methods employed and adherence to quality control and calibration requirements. When the supervisory review is completed the data are approved for reporting to the customer. As noted below, reports cannot be printed from the LIMS until results are approved and only supervisory personnel are granted that authorization by LIMS security measures.

Errors noted in either of these reviews are brought to the attention of the analyst so that the mistakes will not be repeated. If the errors are such that the data are invalidated, the analysis is repeated.

## 8.4 Reporting Analytical Data

All reports except those generated for analyses performed by GC/MS are prepared by the LIMS. In general, three kinds of reports are prepared by ARDL; 1) those in which sample results only appear; 2) those in which sample results and supporting QC measurements appear; and 3) complete data packages which meet all requirements of the USEPA. Reports containing sample results only are standard. Other report formats can be prepared if arrangements are made prior to receipt of samples.

### 8.4.1 Results Only

Most reports generated for customers consist of sample results alone. These reports are prepared in appropriate format by the LIMS. The reports contain information regarding the samples involved (e.g., sample matrix, date, sample collector, sampling point, customer identification for the sample, etc.). The method(s) used to obtain the sample results also appear on the report.

Sample results are reported as follows:

<u>Analytical Value Found</u>	<u>Reported Value to Customer</u>
Up to 0.9999	2 or 3 decimal places* (0.XX or 0.XXX)
1.0 to 9.8999	1 decimal place* (X.X)
10 up	No decimal place* (XX)

\* Dependent upon IDL and CRDL.

### 8.4.2 Results and QC Data

Data submitted includes sample results reported as described above and a tabular summary of the values obtained on spikes, duplicates and other QC samples or analytes.

### 8.4.3 Complete Data Packages

The data are submitted in a format compliant with current USEPA requirements for reporting. These data packages are extensive in nature and are computer generated by special software.

### 8.4.4 Diskette Deliverables

If prior arrangements are made, ARDL has the capability to generate computer readable diskettes which contain all the data appearing in hard copy form as described above. In the past diskettes readable by Word Perfect (Version 5.0) and diskettes in USEPA Format B have been prepared. Diskettes can also be prepared in spreadsheet format, provided specific information regarding the desired format is received from the customer.

## 8.5

### Significant Figures

The term significant figure is defined as the figures of a number that end with the last figure to the right that is not zero or is a zero that is considered to be correct.

A significant figure is a digit (0 to 9) that denotes the amount of the quantity in the particular decimal place in which it stands. A value is made up of significant figures when it contains all digits known to be true and correct and one last digit which is in doubt. Analytical values reported by ARDL contain only significant figures.

The number zero may or may not be a significant figure. ARDL uses the following rules order to determine if a zero is significant:

1. Final zeros after a decimal point are always significant.
2. Zeros before a decimal point with non-zero digits preceding them are significant. With no preceding non-zero digits, a zero before a decimal is not significant.
3. If there are no non-zero digits preceding a decimal point, the zeros after the decimal point but preceding other non-zero digits are not significant - they only serve to locate the decimal point.

Once the number of significant figures has been established for a particular parameter, data resulting from this analysis are reduced according to rounding rules as described in the following section.

## 8.6

### Rounding Rules

Rounding numbers is necessary in all analytical areas and is automatically applied by the limits of all instrumental measurements and glassware. When applied to chemical calculations incorrectly or prematurely, it can have adverse effects.

With the exception noted below, the rounding rules utilized in the laboratory are those employed in Hewlett-Packard calculators. These rules are:

1. If the figure following those to be retained is less than 5, the figure is dropped and the retained figures are unchanged.
2. If the figure following those to be retained is greater than 5, the figure is dropped and the last retained figure raised by 1.
3. If the figure following those to be retained is 5, the figure is dropped and the last retained figure is raised by 1, if it is an odd number. If this digit is an even number it is kept unchanged. (Note: this rule is utilized by ARDL to conform with USEPA rounding specifications.)

4. When adding a series of numbers, the sum is rounded off to the same number of decimal places as the addend with the smallest number of places. The rounding is performed after addition is complete.
5. When subtracting, rounding is performed after the subtraction is complete.
6. When multiplying, all digits are carried through the mathematical operation and the product is rounded. The number with the fewest significant digits determines the rounding position.
7. When dividing, the division is carried out using all digits and the quotient is rounded to the number of significant digits of the divisor or dividend, whichever has the fewer.
8. In chain computations, all digits are carried through all calculations and the final answer is rounded.

**Control Charts**

Stewart control charts are maintained for all quality control data acquired in the course of sample analysis. The charts are reviewed by the Quality Assurance Administrator on a regular basis. They are evaluated with respect to overall conformance to applicable limits and frequencies and also inspected for the appearance of trends which may indicate present or future problems with data quality.

Until early 1992, these charts were kept on diskettes readable by Sigmaplot software (Version 4.02, Jandel Corporation). The data were entered into the system by keyboard. For some parameters, that practice is being continued at present. For quality control data relative to trace metals, most inorganics and a limited number of organics, however, these data are being accumulated in quality control files integral to the PC based LIMS now in use in the laboratory. The data may be reviewed on screen or obtained in hard copy as Stewart charts or tables by individuals with appropriate password authority on the system.

**Blind Control Samples**

At irregular intervals samples of known concentration are logged in as samples received from customers. These samples are either obtained from outside sources or prepared from reagent chemicals in the laboratory. ARDL personnel who may prepare these unknowns are not associated with the sections which will perform the analyses. In any event, details regarding preparation of the unknowns are recorded in a logbook reserved for that purpose. The logbook is held by the Quality Assurance Administrator.

Results of analysis of the samples are reported to the Quality Assurance Administrator. After review of the data, a report is prepared and circulated to cognizant management personnel including the supervisor(s) of the section(s) which performed the analysis. If results so indicate, corrective actions are taken to improve performance on the analysis evaluated. Poor performance by an analytical group or section increases the frequency of audit samples submitted to them.

**Internal Audits**

Audits of analytical group operations are performed on a regular basis by the Quality Assurance Administrator and/or his designee.

**General Audits**

The Quality Assurance Administrator performs a detailed audit of all operations in each analytical group at least annually. This audit consists of close inspection of all records pertaining to preparation of standards, calibration of equipment, logbook entries, maintenance logs and schedules, QC procedures, method selection, adherence to method specifications, sample handling, calculation of results and similar matters. In addition to review of documents and

records, the audit consists of technical discussions with analysts to determine their understanding of the methods in use and the protocols associated with them.

A conference with the analysts and their supervisor closes the audit. That conference includes comment on operations which are in compliance with established guidelines as well as any deviations which have been observed. Actions necessary to correct the deviations are outlined. After these actions have been implemented, the supervisor of the section notifies the Quality Assurance Administrator in writing that his recommendations have been followed.

A written report of the results of the audit is prepared and circulated to cognizant management personnel.

### **9.3.2      Specific Audits**

A number of records (e.g., balance calibrations, sample storage location temperatures, reagent water quality, etc.) are critical to overall laboratory performance. At least monthly, the Quality Assurance Administrator or his designee reviews these records to ensure that; 1) the observations are being made at the proper frequency, 2) appropriate records are on hand, and 3) appropriate corrective actions have been taken when measurements are not within specification. Deviations from requirements for these records are immediately brought to the attention of the supervisor of the group responsible for the records.

The date of the specific audit is noted on a computerized spreadsheet log and a report of the audit results is circulated to cognizant management personnel.

### **9.4      Performance Evaluation Samples**

In addition to analysis of performance evaluation samples associated with the various certifications and approvals cited in Section 9.5, below, ARDL has participated for some years in other programs which involve analysis of performance evaluation samples submitted by outside sources. Currently ARDL participates in the WP, WS and NPDES studies administered by the USEPA.

The WP and WS studies are voluntary. The studies involve analysis of unknown blind water samples for trace metals, inorganics, physical properties, pesticides, PCB's and volatile organics on a semi-annual basis. Results submitted are not scored officially. ARDL's submitted results, however, compare favorably (85-95% correct) with true values and acceptance windows published after all results have been evaluated. ARDL has participated in the WS study since November, 1989 and in the WP study since January, 1990.

Because the results from ARDL analysis of some customer samples are submitted as a condition of that customer's NPDES permit, participation in the NPDES study is required. The analyses required vary, dependent on the parameters which the customers must monitor as a condition of their permit. A report of PES results is issued to the customer by the USEPA and no scores are issued. If an erroneous result has been submitted, ARDL reanalyzes the sample or its replacement.

**Certification and Approvals**

ARDL is certified for analysis of an exceptionally wide range of inorganic and organic analytes in soil and water samples under Defense Environmental Restoration Program administered by the U.S. Army Corps of Engineers. Certification is renewed biannually following successful analysis of performance evaluation samples and a detailed on-site audit of laboratory procedures by Corps of Engineers personnel. This certification has been in place since 1988. The certification extends to approval of ARDL as an analytical laboratory by other arms of the Defense Department.

ARDL is certified for analysis of trace metals and inorganics in water by the Oklahoma Water Resources Board. Certification is renewed annually after successful analysis of performance evaluation samples. The certification has been in place since 1989.

ARDL is certified for analysis of trace metals, inorganics and organics in drinking water by the Illinois Environmental Protection Agency. Certification is renewed annually after successful analysis of performance evaluation samples and a detailed on-site audit of laboratory procedures by agency personnel. The certification has been in place since 1988.

ARDL is an approved laboratory in the contract laboratory programs of the Illinois Environmental Protection Agency and the Indiana Department of Environmental Management. Approval is granted after a successful on-site audit of laboratory procedures by personnel from the agencies and is contingent upon successful analysis of performance evaluation samples received from other sources.

ARDL is an approved laboratory in the USEPA SAS (Special Analytical Sample) program and regularly analyzes samples received from the USEPA Sample Management Office.



**PREVENTATIVE MAINTENANCE, PROCEDURES AND SCHEDULES**

Preventative maintenance procedures are outlined in Appendix H.

## 11.0 CORRECTIVE ACTIONS

### 11.1 Out of Limit QC Results

As part of the preliminary steps taken, the out of limit QC sample may be reanalyzed as described in Section 7, above. A review of logbook entries relative to sample weight or volume, dilutions, calculations, etc., of the spurious value is also performed. If no calculation errors are identified, further evaluation of instrument function is performed. Upon completion of these initial checks the analyst informs the supervisor of the problem and of the results of the preliminary investigations. The supervisor evaluates the data acquired and decides on the next steps to be taken.

As warranted by the situation, the analyst and supervisor (and other laboratory staff members as required) begin an effort to identify the cause of the problem. Details of the investigation are recorded in the analyst's logbook. Based on the results of that effort the necessary corrective actions are taken and the out-of-control analysis is repeated to demonstrate that the problems has been resolved.

A brief memorandum reporting the essentials of the problem and the corrective action taken is submitted to the Quality Assurance Officer and cognizant management personnel.

All analytical results obtained between the most recent acceptable QC value and the out-of-control value are disregarded and the analyses are repeated. Sample analysis is then continued in normal fashion.

### 11.2 Unusual Occurrences

When a laboratory accident (e.g., dropped sample, data loss through power failure, etc.) occurs the analyst notes the event in the logbook and informs the supervisor. Appropriate action is taken immediately based on the best judgment of the supervisor. Notations are made in the logbook regarding the corrective actions taken and the notations initialed and dated. If the occurrence is of such magnitude that it impacts on overall laboratory operations the Technical Services Manager and General Laboratory Manager are informed. A brief written report of the occurrence and the steps taken to correct the problem may be required by either of those individuals.

**QUALITY ASSURANCE REPORTS TO MANAGEMENT**

In addition to the reports made relative to corrective actions, internal audits, blind quality control samples and other activities. Summaries of QC data are prepared in temporary data files in the LIMS by the Quality Assurance Officer. Notice is given to cognizent supervisors that the files are available for review or screen. In the event that the frequency of outliers has been unusually high for a given analysis or in analyses from a specific analytical section, the Quality Assurance Officer prepares a written report describing the situation and outlining the corrective actions being taken.

## **APPENDIX A**

### **Standard Operating Procedures**

**TABLE 1**  
**VOLATILE STANDARD STOCK SOLUTIONS AND REAGENTS**

**Methanol:** Burdick & Jackson. Purge and trap grade. Stored at room temperature in the GC/MS room, free from other solvent vapors.

**4-Bromofluorobenzene (BFB):** Aldrich cat. no. B6,720-1. Stored in refrigerator at 4°C.

**m-Xylene:** Eastman. Stored at room temperature.

**p-Xylene:** Fisher. Certified grade. Stored at room temperature.

**Purgeable A:** Supelco Cat. No. 4-8851 contains 200 ug/ml each of the following solvents in methanol;

Methylene chloride	Carbon tetrachloride
Dibromochloromethane	Trichlorofluoromethane
1,2-Dichloropropane	2-Chloroethyl vinyl ether
1,1-Dichloroethene	Trichloroethene
Tetrachloroethene	1,1-Dichloroethane
1,1,2-Trichloroethane	Chlorobenzene
Chloroform	

**Purgeable B:** Supelco Cat. No. 4-8852 contains 200 ug/ml of each of the following solvents in methanol.

trans-1,2-Dichloroethene	Benzene
Bromoform	1,2-Dichloroethane
trans-1,3-Dichloropropene*	Toluene
1,1,1-Trichloroethane	cis-1,3-Dichloropropene*
Ethyl benzene	1,1,2,2-Tetrachloroethane
Bromodichloromethane	

**Purgeable C:** Supelco Cat. No. 4-8853 contains 200 ug/ml of each of the following solvents in methanol:

Chloromethane	Bromomethane
Vinyl chloride	Chloroethane

**HSL Volatiles:** Supelco Cat. No. 4-8920 contains 2000 ug/ml of each of the following solvents in methanol:

Acetone	Styrene
Carbon Disulfide	Vinyl acetate
2-Hexanone	2-Butanone
4-Methyl-2-pentanone	o-Xylene

**Internal Standard:** Supelco Cat. No. 4-8835 contains 1000 ug/ml each of the following solvents in methanol;

Bromochloromethane	d <sub>5</sub> -Chlorobenzene
1,4-Difluorobenzene	

**Surrogate Standard:** Supelco Cat. No. 4-8876 contains 250 ug/ml each of the following solvents in methanol;

d <sub>4</sub> -1,2-Dichloroethane	d <sub>8</sub> -Toluene
4-bromofluorobenzene	

\*Dichloropropenes in Purgeable B are at concentrations of 400 ug/ml

#### 2.2.1.1.5 Matrix Spike

Using appropriate dilutions, a working stock solution containing 25 ng/ul of each of the compounds 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene and benzene is prepared as required from the certified standard stocks described in Table 1. The diluted standard is stored at approximately -10°C until needed for use. The contents, date of preparation and analyst name are clearly marked on the container label.

#### 2.2.1.2 Daily Working Calibration Standards

Aqueous calibration standards are prepared daily in the range of concentrations required. The procedure followed is outlined below.

- A 5 ml gastight syringe fitted with a valved Leurlok adapter is filled to volume with reagent water and the plunger withdrawn slightly.
- The valve is opened and a 5 ul aliquot of the internal standard working stock is added to the water in the syringe through the valve bore. The valve is closed.
- The valve is opened and a 5 ul aliquot of the surrogate working stock is added to the water in the syringe through the valve bore. The valve is closed.
- The valve is opened and appropriate volumes of the working calibration standard stock is added through the bore to produce the desired concentration in the daily working standard. The valve is closed after each addition.
- After addition of the last aliquot of calibration standard stock, the daily standard is ready for use. To prepare a 50 ppb calibration standard, 5 ul of each working stock is added. To prepare a 100 ppb calibration standard, 10 ul of each working stock is used. 40 ul.

#### 2.2.2 Semi-Volatile Standards (GC/MS)

##### 2.2.2.1 Reagents and Standard Stock Solutions

The reagents and standard stock solutions used for semivolatile analyses are described in detail in Table 2. The methylene chloride is stored at room temperature. All other materials are stored at 4°C.

##### 2.2.2.2 Working Standards and Reagents

Base/neutral working standards are prepared on an as needed basis (usually every two to six weeks) as described below. In all cases the working standards are stored at 4°C. DFTPP working reagent is prepared fresh every three months and is stored at 4°C. Working standards and reagents are stored in properly labeled clean glass screw cap vials fitted with Teflon lined caps.

**TABLE 2**  
**SEMI-VOLATILE STANDARD STOCK SOLUTIONS AND REAGENTS**

**Methylene Chloride:** J.T. Baker Resi-Analyzed Grade.

**Decafluorotriphenylphosphine (DFTPP):** Supelco Cat. No. 4-8724.25 mg/ml in methylene chloride.

**Base-Neutral Mix 1:** Supelco Cat. No. 4-8900 contains 2000 ug/ml each of the following solvents in methylene chloride;

bis(2-Chloroethoxy) Methane	N-nitrosodimethylamine
N-nitrosodi-n-propylamine	bis(2-Ethylhexyl)phthalate
Di-n-butylphthalate	4-Bromophenyl phenyl ether
bis(2-Chloroethyl)ether	N-Nitrosodiphenylamine
Butylbenzylphthalate	Diethylphthalate
Dimethylphthalate	bis(2-chloroisopropyl)ether
4-chlorophenylphenylether	Di-n-octylphthalate

**Base-Neutral Mix 1:** Supelco Cat. No. 4-8901 contains 2000 ug/ml each of the following solvents in methylene chloride;

Azobenzene	Hexachlorobutadiene
Hexachlorobenzene	1,2-Dichlorobenzene
Hexachloroethane	Hexachlorocyclopentadiene
1,3-Dichlorobenzene	Nitrobenzene
Isophorone	1,4-Dichlorobenzene
2-Chloronaphthalene	1,2,4-Trichlorobenzene
2,6-Dinitrotoluene	2,4-Dinitrotoluene

**Phenols Mix:** Supelco Cat. No. 4-8904 contains 2000 ug/ml each of the following solvents in methylene chloride;

4-Chloro-3-methylphenol	Pentachlorophenol
2,4-Dichlorophenol	2,4,6-Trichlorophenol
4-Nitrophenol	2,4-Dinitrophenol
2-Chlorophenol	Phenol
2-Nitrophenol	2,4-Dimethyl phenol
2-Methyl-4,6-dinitrophenol	

**PAH Mix:** Supelco Cat. No. 4-8905 contains 2000 ug/ml each of the following solvents in 50:50 methylene chloride:benzene;

Acenaphthene	Fluorene
Benzo(b)fluoranthene	Acenaphthylene
Fluoranthene	Anthracene
Ideno(1,2,3-cd)pyrene	Dibenzo(a,h)anthracene
Benzo(a)pyrene	Phenanthrene
Benzo(g,h,i)perylene	Pyrene
Chrysene	Naphthalene
Benzo(a) anthracene	Benzo(k)fluoranthene

**TABLE 2**  
(Continued)

**SEMI-VOLATILE STANDARD STOCK SOLUTIONS AND REAGENTS**

**Benzidines Mix:** Supelco Cat. No. 4-8906 contains 2000 ug/ml each of the following solvents in methanol;

Benzidine	3,3'-Dichlorobenzidine
-----------	------------------------

**Hazardous Substances Mix 1:** Supelco Cat. No. 4-8907 contains 2000 ug/ml each of the following solvents in methylene chloride;

Benzonic acid	2-Methyl phenol
2,4,5-Trichlorophenol	4-Methyl phenol

**Hazardous Materials Mix 2:** Supelco Cat. No. 4-8908 contains 2000 ug/ml each of the following solvents in Methylene chloride;

Aniline	3-Nitroaniline
2-Nitroaniline	4-Chloroaniline
Benzyl alcohol	4-Nitroaniline
2-Methylnaphthalene	Dibenzofuran

**Internal Standard Mix:** Supelco Cat. No. 4-8902 contains 2000 ug/ml each of the following solvents in methylene chloride;

d <sub>10</sub> -Acenaphthene	d <sub>8</sub> -Naphthalene
d <sub>12</sub> -chrysene	d <sub>4</sub> -1,4-Dichlorobenzene
d <sub>10</sub> -Phenanthrene	d <sub>12</sub> -perylene

**Acid Surrogate Mix:** Supelco Cat. No. 4-8875 contains 2000 ug/ml each of the following solvents in methanol;

2-Fluorophenol	d <sub>6</sub> -Phenol
2,4,6-Tribromophenol	

**Base-Neutral Surrogate Mix:** Supelco Cat. No. 4-8925 contains 1000 ug/ml each of the following solvents in methylene chloride;

d <sub>5</sub> -Nitrobenzene	2-Fluorobiphenyl
d <sub>14</sub> -4-Terphenyl	



1. DFTPP 25 ng/ul solution is made up by diluting 10 ul of DFTPP stock (25 mg/ml) to 20 ml with methylene chloride.
2. Standard Solutions - To prepare working standard solutions for calibration of the GC/MS for acid/base neutral analyses the following volumes of each of the standard mixes noted in Table 2 are added to clean glass vials. The volumes added are:

10 ng/ul working standard - 5 ul of each of the mixed standards identified in Table 2.

25 ng/ul - 12.5 ul each, as above

40 ng/ul - 20 ul each, as above

60 ng/ul - 30 ul each, as above

80 ng/ul - 40 ul each, as above

To each of the vials, aliquots of 10 ul internal standard mix, 50 ul acid surrogate mix and 50 ul base/neutral surrogate mix are added. On final dilution these volumes will result in an internal standard concentration 20 ng/ul, an acid surrogate concentration 100 ng/ul and a base/neutral surrogate concentration 50 ng/ul. These volumes are made to 1.0 ml with methylene chloride to provide the working standards required.

### 2.2.3 Pesticide Standards

Five concentrations of standards are prepared from a working stock solution. These working stocks and the resultant standards are prepared by laboratory personnel using stocks of pure analytes obtained from various vendors. The vendors certify, identify purity and concentration of analyte involved.

To prepare a working stock solution, the following volumes of each of the standard mixes noted in Table 3 are added to a clean volumetric flask:

100 ul of pesticide mix;

1 ml of dibutylchlorendate solution; and

1 ml of 2,4,5,6-tetrachloro-m-xylene solution.

The flask contents are brought to volume with a suitable solvent (isooctane, acetone, etc.) to produce a solution containing 2.0 ng of each analyte per ml.

Dilutions are made from the working stock in 10 ml. volumetric flasks to obtain concentrations of 0.10, 0.20, 0.50, 1.0 and 2.0 ng/ml of each analyte for most applications. On occasion, however, lower standard concentrations are prepared from these same stocks by making adjustments in the volumes taken for dilution.

TABLE 3

PESTICIDE INDIVIDUAL STANDARD STOCK MIXES

Pesticide Mixture: Supelco Catalog 4-8913 (2 mg each analyte/ml of 1:1:toluene:hexane)

Aldrin	Methoxychlor
alpha-BHC	Endosulfan I
beta-BHC	Endosulfan II
delta-BHC	Endosulfan Sulfate
gamma-BHC (Lindane)	Endrin
4,4'-DDD	Endrin Aldehyde
4,4'-DDE	Endrin Ketone
4,4'-DDT	Heptachlor
Dieldrin	Heptachlor Epoxide

Surrogate Solutions:

Supelco 4-8317M contains 200 ug/ml of 2,4,5,6-tetrachloro-m-xylene in methanol.

Supelco Quote 3405 contains 200 ug/ml dibutylchloroendate in acetonitrile.

Degradation Standard:

Supelco 4-8962M contains 200 ug/ml Aldrin in isooctane.

Supelco 4-8980M contains 200 ug/ml 4,4'-DDT in isooctane.

Supelco 4-8976 contains 200 ug/ml Endrin in isooctane.

In addition to calibration standards, a degradation check solution is also prepared. The solution contains the compounds Aldrin, Endrin, 4,4'-DDT and dibutylchlorendate at a concentration of 0.5 ng/ml for Aldrin and 1.0 ng/ml for the other components. Using the certified stocks identified above, 0.50 ml of Aldrin solution and 1.0 ml of the solutions of the other components are made to 100 ml volume in isooctane or acetone. The solution is then diluted 1:1 with additional solvent to obtain the degradation check working solution.

All working standards are stored in glass screwcap vials at 4°C. The bottles are clearly labeled with the name of the preparation, date of preparation and logbook reference.

#### **2.2.4 PCB and Toxaphene Standards**

PCB and Toxaphene stock standards are prepared separately at 5000 ng/ul from neat standards or vendor certified solutions. Separate working stocks of each multicomponent analyte are prepared by making appropriate dilutions of the vendor stock. These stocks are used to prepare working standards, as required.

All working stocks and standards are stored in glass screwcap vials at 4°C. The bottles are clearly labeled with the name of the preparation, date of preparation, analyte concentration and logbook reference.

### **3.0 TRACEABILITY OF STANDARDS**

Analytical standards at ARDL are purchased already prepared from various scientific supply organizations or prepared in the laboratory from reagent chemicals. Prepared standards are certified by those suppliers regarding identity, purity and concentration. Reagent chemicals used to prepare standard solutions are certified regarding actual lot analysis for identity and purity according to specifications of the American Chemical Society. Documentation of the receipt, storage, preparation and use of standards consists of the following:

1. Inventories of standard materials are routinely maintained by an analyst from organic section and an analyst from the inorganic section. The inventories show material lot number, date of receipt and, where appropriate, an expiration date. It is policy to replace standards that have been in the laboratory longer than six months even though a vendor may indicate a longer shelf life. The inventories also indicate the date a specific item was withdrawn from stock for use and the individual who withdrew it.
2. Dated entries are made in the analyst's logbook relative to the chemical identity and lot number of the standard used and the procedures employed in preparation of the standard stock solution as well as any working standards prepared. Working standards are assigned a batch number when their stability permits use over an extended period of time.

3. Dated entries are made in the analyst's logbook relative to identity and lot (or batch) number of the standards used during analysis of sample lots or sublots.

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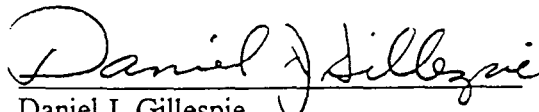
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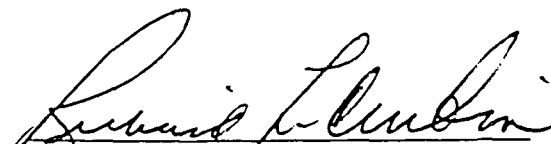
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**INSTRUMENT RECORDS AND LOGBOOKS**

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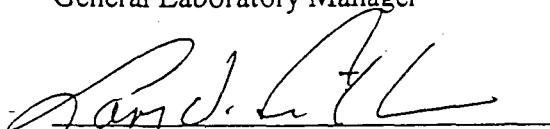
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Daniel J. Gillespie  
Technical Services Manager

11/18/92

  
Richard L. Curtin  
General Laboratory Manager

11/18/92

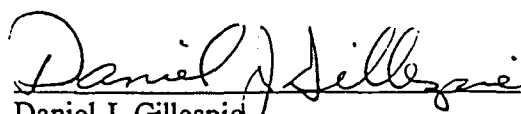
  
L. V. Gibbons, Ph.D.  
President and Laboratory Director

11/18/92

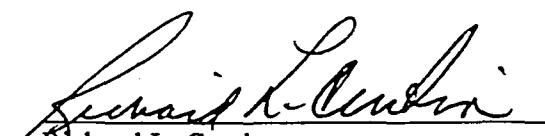
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**GLASSWARE CLEANING PROCEDURES**

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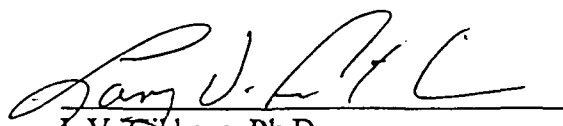
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## 1.0 ROUTINE GLASSWARE

Good laboratory practice dictates that all vessels should be rinsed with tap water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove at a later time.

Routine glassware at ARDL is cleaned by the following protocol:

1. Rinse with tap water.
2. Wash in a hot, 3% solution of Alconox in tap water (a brush is used to ensure adequate cleaning).
3. Rinse well with warm tap water.
4. Rinse well with deionized water.
5. Allow to air dry.

## 2.0 TRACE METAL GLASSWARE

Glassware for trace metal analysis is cleaned as in 1.0 above, then washed with 50%  $\text{HNO}_3$  followed with deionized water (at least 3). Glassware is then given a final rinse with reagent water. This glassware is kept separate and used only for metal analysis.

## 3.0 NUTRIENT ANALYSIS GLASSWARE

Glassware used in nutrient analysis is first cleaned per Section 1.0 above. Then 50%  $\text{HCl}$  is added to the glassware and the contents boiled for at least 15 minutes. The  $\text{HCl}$  should reflux down the inside of the glassware. After the vessels have cooled, they are rinsed with tap water several times, then with deionized water several times and finally rinsed with ammonia free distilled water. After air drying, this glassware is segregated and maintained for nutrient analyses only.

## 4.0 TRACE ORGANIC GLASSWARE

### 4.1 Sampling Containers

Trace organic analysis glassware is first cleaned as outlined in Section 1.0 above. This glassware is cleaned with a 15 minute chromic acid wash. After the chromic acid wash, the glassware is rinsed several times with tap water and finally a deionized water rinse.

Following this water rinse step, the vessels are subjected to a warm 50% HNO<sub>3</sub> rinse/wash. The glassware is again well rinsed with deionized water (no tap water) and followed with at least three rinses of distilled water.

The final step is rinsing of the glassware with HPLC grade methylene chloride and oven drying at 400°C for one (1) hour. Caps are washed as outlined in Section 10.1 above, rinsed with tap water, deionized water, distilled water, methylene chloride and dried at 6°C (Vacuum) for 12 hours.

The ovens are turned off and when the glassware has cooled, it is sealed (cap or aluminum foil) and removed to covered storage for future use.

#### 4.2 Analytical Containers

Glassware for trace organic analysis is first cleaned as follows:

1. Tap water rinse.
2. Wash in hot Alconox soap solution in tap water.
3. Rinse well with warm tap water
4. Rinse well with de-ionized water.

Following the above procedure, a chromic acid rinse is used to remove all traces of residual organic material. Extended periods in the chromic acid wash may be required for extremely dirty glassware.

Following the chromic acid rinse, the glassware is rinsed several times with tap water to remove all traces of the chromic acid. The glassware is then rinsed again with de-ionized water. Next, the glassware is triple rinsed with acetone to remove all traces of water. This is followed by a final triple rinse with methylene chloride. If the glassware is to be used for pesticide/PCB analysis, the methylene chloride is then followed by a hexane triple rinse.

Following this cleaning procedure, the glassware is allowed to air dry, then placed in the appropriate cabinet(s) for storage until use.

All solvents used in the cleaning of trace organic glassware must be pesticide grade solvents or their equivalent.

Following use in analytical procedures, all trace organic glassware must be rinsed with last solvent used in the analysis to remove any residual sample components from the glassware prior to these cleaning procedures.

PIPETS

Pipets are allowed to soak in "RBS" (surfactant) until a sufficient quantity has accumulated for washing. Washing is accomplished by repeatedly raising and lowering the pipet basket in the RBS solution. This is repeated for at least five (5) repetitions at which time the pipets are placed in the pipet washer which automatically fills and empties the pipets with tap water (hot). This is allowed to continue for 15 to 30 minutes. The pipets are then rinsed with three (3) different changes of deionized water. The cleaned pipets are oven dried at 200°F overnight.

The oven is allowed to cool, pipets are removed, sorted and stored in proper covered drawers.

SPECIAL CLEANING

Glassware that is non-cleanable by any of the above procedures is cleaned by treatment with chromic acid cleaning solution. This could be burets, pipets, volumetrics or any other type of glassware.

The cleaning procedure is to fill the container with chromic acid and allow it to react for at least 15 minutes. If heat can be applied, the chromic acid solution should be heated for better, faster cleaning action. After the chromic acid has reacted, it is placed back into its stock container and the vessel being cleaned is rinsed with tap water followed with deionized water. All traces of chromic acid must be removed!

Chromic acid solution is prepared by adding 1 L of concentrated  $H_2SO_4$  slowly with stirring to a 35 ml saturated sodium dichromate solution or by adding one vial of Chromerge (Fisher Scientific) to a 9 lb. bottle of concentrated  $H_2SO_4$  and mixing.

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## 1.0 INSTRUMENT RECORDS AND LOGBOOKS

Bound laboratory logbooks with pages numbered in ascending order from front to back are utilized in the laboratory. The logbooks are numbered sequentially and are issued by number to specific analysts for recording sample analysis or to analytical groups for maintaining various daily logs. Entries in logbooks are made in chronological order.

Analysis of samples for a given parameter are scheduled in blocks of convenient size. In practice, the most appropriate block size is approximately 20 samples. Data for this number along with the requisite QC analyses, calibrations and exemplar calculations are about the maximum amount of information which can conveniently fit in the space available.

### 1.1 Instrument Records

#### 1.1.1 Maintenance Logbooks

ARDL maintains laboratory notebooks on each major instrument in the laboratory. Pertinent information on routine maintenance, breakdowns, and special service are recorded in each notebook. Each specific item is dated by the analyst. Operating manuals are kept near the instrument or in ARDL's filing system to allow quick retrieval in case of emergency.

A copy of a page from one of the maintenance logbooks appears in Figure 4.

#### 1.1.2 Run Logs

Detailed logs of all major instrument analyses are maintained in accordance with standard practices. These logs contain records of those data pertinent to the method being employed. At a minimum this will include:

- a. date and time of the analysis.
- b. calibration data or reference thereto.
- c. identification of samples analyzed.
- d. dated signature of the analyst.

Copies of pages from run logs appear in Figures 1, 2 and 3.

## FIGURE 1

## GC/MS Maintenance Log

4/24/92 (HP3)

- In MTUNE gives error message of amplifier trouble. check cables on GTO board. Am going to take source out & check it.
- Source looked O.K. Put back together & checked for air. Had air but did spectrum scan O.K.

4/27/92 (HP3)

- Same error message

XXXXTurning MS on . .

MANU/

The status returned from the scanning interface was not in the range 20 thru 98. This is either due to:

1. The communications between RTE and the scanning interface are not synchronized. Try running the program once again.
2. The data transmitted from the scanning interface is not reaching RTE properly. This may be due to a faulty cable, faulty GPIO board, or other scan box hardware problems.

Clearing interface IO . . DONE

- call ARCS
- tried self tests; scan box, DMA Scan box; both no error
- mass spec self test no error
- Setting RPPA There is a probl w/ mass filter electronics (Bit 7) Parity fault has occurred.
- Mike Ac to be here 4/29 A.M.

Continued on Page \_\_\_\_\_

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Signed \_\_\_\_\_

Date \_\_\_\_\_

Signed \_\_\_\_\_

Date \_\_\_\_\_

PROJECT

12/10/92FIGURE 2  
GC/MS Run Log

Sequence File: VMPO10::SC

Tune File: MTHP4

CR List: D5,D4

CR Cut-off: 1500 blocks.

Number of samples: 15		Sample Bottle	Sample Size	Report Quant	Data Archive	ID Archive	Output Archive
Samp No.	Data File Method File	Sample Name Misc Data					Quant ID Quant Output
001)	>R2962	9252-7 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASLW308WA		Dil=1.00000			^R2962::D1
4	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
002)	>R2963	9252-8 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASLW301WA		Dil=1.00000			^R2963::D1
5	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
003)	>R2964	9252-9 Actual LEU ↑	0	5 Y E N N N			IDW4MA::SC RECUR
	VOAME2	HP-4 5ML 92ASDE807WA		Dil=1.00000			^R2964::D1 1/10
6	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
004)	>R2965	9252-10 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW250WA		Dil=1.00000			^R2965::D1
7	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
005)	>R2966	9253-1 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW201WA		Dil=1.00000			^R2966::D1
8	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
006)	>R2967	9253-2 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW211WA		Dil=1.00000			^R2967::D1
9	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
007)	>R2968	9253-3 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW221WA		Dil=1.00000			^R2968::D1
12	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
008)	>R2969	9253-4 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW231WA		Dil=1.00000			^R2969::D1
13	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
009)	>R2970	9253-5 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW202WA		Dil=1.00000			^R2970::D1
14	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
010)	>R2971	9253-6 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW209WA		Dil=1.00000			^R2971::D1
15	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
011)	>R2972	9253-7 OK	0	5 Y E N N N			IDW4MA::SC
	VOAME2	HP-4 5ML 92ASMW212WA		Dil=1.00000			^R2972::D1
16	REPRT OPTIONS: Label Meth:1, B option, Scale time: 3.000						
	REPRT OPTIONS: 3 page TIC, Omit types:IS						
012)	>R2973	9253-8 OK	0	5 Y E N N N			IDW4MA::SC

2

Continued on Page \_\_\_\_\_

Read and Understood By \_\_\_\_\_

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Date \_\_\_\_\_

Signed \_\_\_\_\_

Date \_\_\_\_\_



PROJECT 11/11/92

Continued From Page \_\_\_\_\_

BFB - 9001335				8020 MEX - 9001217				Tape - GC DATA 59				Volume - HPS 9114			
1)	040PPB	STD	OK					HPS 18.90 R							
2)	BLANK	10 $\mu$ L BFB	OK									91			
3)	<del>9271</del> 9269-18	10 $\mu$ L of 200 DL (100,000 DL)	OK									92			
4)	9271-9	10 $\mu$ L / 5 mL	500 DL	CLEAN NOT NEEDED				OK				93			
5)	9271-10	10 $\mu$ L / 5 mL	500 DL									94			

Sequence file Not named yet Date = 11-11-1992 Time = 12:52:00

PLOT #	DATA FILE	SAMPLE NAME	METHOD	SAMPLE WEIGHT	AMOUNT INJECTED	DILUTION FACTOR	INTERNAL STD AMT	CAL
9	1 HPSN11.01R 95	6288-1 5ML OK	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
10	2 HPSN11.02R 96	CLEANOUT CLEAN	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
11	3 HPSN11.03R 97	6288-2 5ML OK	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
12	4 HPSN11.04R 98	6288-3 1/100 OK has been Conf	F:\CP1\HP5P\HP5	1.000	1.000	100.000	1.000	0
13	5 HPSN11.05R 99	6290-1 5ML OK CONFON AS	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
6	6 HPSN11.06R 00	6287-2 1G HGLBKG Return 100 DL	F:\CP1\HP5P\HP5	0.910	1.000	5.000	1.000	0
7	7 HPSN11.07R 01	6287-3 1G OK ALREADY CONF	F:\CP1\HP5P\HP5	0.920	1.000	5.000	1.000	0
8	8 HPSN11.08R 02	6287-4 5G BFB Return 5g	F:\CP1\HP5P\HP5	0.640	1.000	1.000	1.000	0
15	9 HPSN11.09R 03	CLEANOUT CLEAN	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
16	10 HPSN11.10R 04	CLEANOUT	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
11	11 HPSN11.11R 05	040PPB STD OK	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0
12	12 HPSN11.12R 06	BLANK OK	F:\CP1\HP5P\HP5	1.000	1.000	1.000	1.000	0

Continued on Page \_\_\_\_\_

Read and Understood By

Signed

Date

Signed

Date

FIGURE 4  
AA Run Log

Notebook No. 764

76

PROJECT \_\_\_\_\_

Continued From Page \_\_\_\_\_

DATE	INSTR	ELEMENT	SAMPLES ANALYZED	VALID	OP
11-11-92	4000	As	all DIS 6283-(1-5) + A Sp on all 6288(1,2) + A Sp on all 6289-1,10,2 + A Sp on all 6291(1-5) + A Sp on all 6299-1,2,3,30,4 + A Sp on all 6300-(1-4) + A Sp on all 6301-1 + A Sp on all	all got "W" flag	1AA ↓
11-12-92	AA20	Hg	113799-1,1MS,1MSD 2-1,1DUP 12-26 113789-5,5MS,5MSD 6,7,8,9 TOLP 113790-5,5MS,5MSD 7,8 TOLP 113799-11,11MS,11MSD 16,18,19 TOLP 6286-1,1MS,1DUP TOLP 6287-2,2DUP,2DUP, +4 TOLP 5417-1,1MS,1MSD 2-10 6298-1,10,1Sp + 2 TOT 6299-1,10,1Sp,2,3,4 TOT 6299-1,10,1Sp,2,3,4 DISS 6301-1,10,1Sp TOT 6301-1,10,1Sp DISS 113807-2+3 113812-2+5	all valid	DJR
11-12-92	AA20	Pb	6260-1,2,12,14 - all out of Calibration range 6260-3,3DUP,4,4,7,7DUP,14 needs ex Reso 8,10,11,13 6261 1-6 - all out of calibration	6260E7 15 out - may need MS4	DJR
11-13-92	AA20	Pb	9271-1,2,3,4,4MS,4MSD 5-10 all ran msa	all valid	DJR

Continued on Page \_\_\_\_\_

Read and Understood By

Signed \_\_\_\_\_

Date \_\_\_\_\_

Signed \_\_\_\_\_

Date \_\_\_\_\_

11/17/92

**Analytical Logbooks**

ARDL requires that all data be recorded in ink in bound laboratory notebooks. The lab notebooks are numbered and assigned to personnel as needed. At a minimum, ARDL requires the following information be recorded in the analyst's notebook:

1. Date
2. Sample ID number
3. Measured parameter
4. List of samples, standards and blanks
5. Analytical details:
  - a. Calibration data including identification of standards, preparation of working standards or reference to logbook entries showing that preparation, instrument readings, regression equation calculations.
  - b. Spiking levels.
  - c. Notations regarding all dilutions employed.
  - d. Exemplar calculations.
6. Reported data values.
7. Date and signature of analyst.
8. Date and signature of witness.

ARDL archives completed lab books indefinitely.

Copies of exemplar pages from one of these logbooks appear in Figure 5.

### 1.3 Sample Preparation Logbooks

Preparation of both inorganic and organic samples for analysis is documented in separate notebooks. Records are maintained as noted in Section 1.2, above. Specifically the information included in sample preparation logbooks must be the following:

- Date and analyst initials
- Sample ID Number
- Sample preparation method
- Sample weight or volume
- Reagents, surrogates or spikes added including weight or volume, lot number, concentration, etc., for each
- Final dilution volume of prepared sample

Copies of exemplar pages from logbooks dealing with both organic and inorganic sample preparations appear in Figures 6 and 7.

### 2.4 Error Corrections

All logbook entries are recorded in ink. Erroneous entries are corrected by striking a single line through the mistake and recording the proper value above, below or immediately beside the original value in an unambiguous way. All corrections are dated and initialed. In no case is the original entry obliterated.

FIGURE 5

## Analyst Logbook Page

Notebook No. 1075

3

PROJECT Total SolidsContinued From Page 4

Sample	TH	TWT (g)	TWT + Sample (g)	105° WT	conc %	
113790-1	25	1.00	7.52	6.11	78.4	
113790-2	26	1.00	7.72	6.87	87.4	
113790-3	27	1.00	8.06	7.15	87.1	
113790-4	28	1.00	5.39	4.49	79.5	
113790-5	29	1.00	5.19	4.81	90.9	
113790-6	30	1.00	4.60	4.05	84.7	
113790-7	31	1.00	9.38	8.07	84.4	
113790-8	32	1.00	10.00	8.24	80.4	0.25% RPD
113790-8 (dup)	33	1.00	10.06	8.23	79.8	
113789-1	34	1.00	8.07	6.23	74.0	
113789-2	35	1.00	6.15	5.17	81.0	
113789-3	36	1.00	9.15	6.97	73.3	
113789-4	37	1.00	6.63	5.40	78.2	
113789-5	38	1.01	5.30	4.74	86.9	2.21% RPD
113789-5 (dup)	39	1.00	5.32	4.67	85.0	
113789-6	40	1.00	7.02	5.86	80.7	
113789-7	41	1.01	5.99	5.19	83.9	
113789-8	42	1.01	4.27	3.53	77.3	
113789-9	43	1.01	6.31	5.22	79.4	
113789-10	44	1.01	7.22	6.61	89.5	
113789-11	45	1.01	4.94	4.25	82.4	
113789-12	46	1.01	8.14	5.99	69.8	
113789-13	47	1.01	7.86	6.63	82.0	
113789-14	48	1.01	8.01	6.55	78.9	
113789-15	49	1.01	4.18	3.53	79.5	0.25% RPD
113789-15 (dup)	50	1.45	4.25	3.58	79.3	
113789-16	51	1.01	5.43	4.52	79.4	
113789-17	52	1.01	9.83	7.65	75.3	
113789-18	53	1.01	5.97	4.84	77.2	
113789-19	54	1.01	6.20	5.24	81.5	
113789-20	55	1.01	6.49	4.62	68.9	

Continued on Page 4

Read and Understood By

Larry Hager  
Signed11-2-92  
Date[Signature]  
Signed11/2/92  
Date

PROJECT

9251

Organic Sample Preparation

Continued From Page

Logbook Page

B 2442

BNA (CLP)

Sample ID #	Wt	PH	Vf	Date
Blk ①	—	—	0.5 ml	10/15/92
Blk ②	—	—		
Spk Blk	—	—		
9251 - 1	30.00 g	6.5		
- 3ms	30.00	6.5		
- 3msD	30.00	6.5		
- 2	29.98	6.5		
- 3	29.98	6.5		
- 4	29.97	6.8		
- 5	29.98	6.8		
- 6	29.98	6.4		Spikes 11K
- 7	29.96	6.7		Witness 2M
- 8	29.98	6.8		
- 9	30.00	6.8		
- 10	30.00	6.7		
- 11	30.02	6.9		
- 12	30.02	6.4		
- 13	29.98	6.8		
- 14	30.00	6.3		
- 15	30.00	6.3		
- 16	30.02	6.3		
- 17	30.00	6.9		
- 18	30.00	6.2		

All samples have 50ul of Surrogate A 20035  
 MS & MSD & Spk Blk 50ul of Spike A 19123  
 BN 20006  
 BN 19072

Continued on Page

Read and Understood By

Norman Jackson  
 Signed

10/16/92  
 Date

Steve Newcomb  
 Signed

10/17/92  
 Date

FIGURE 7

Inorganic Sample Preparation  
Logbook Page

58

PROJECT

Notebook No. 1070

Continued From Page

Reference No. 622

ARDL, Inc.

## METALS PREPARATION LOG

Contract: BW4CEMethod: SW846Date: 11-12-92Analyst: D. Upcraft

ARDL Sample No.	Initial Weight (gram/vol)	Final Volume (ml)
PB1	100ml	100ml
PB2		
LCSF		
LCSF TEST	✓	
6264 -1	1.01	
-2	1.02	
-3	1.02	
-4	1.02	
-5	1.01	
-6	1.01	
6266 -1	1.10	
6270 -1	1.05	
-2	1.07	
-3	1.01	
-4	1.00	
-5	1.00	
-6	1.05	
-6 DUP	1.05	
-6 MS	1.05	
-7	1.00	
-8	1.05	
-9	1.02	
6264 -4 DUP	1.02	✓

QC Solution	Mls Added	Solution Used	Reference # (Book #-Pg #)	Spk By Witness
Spike F (GFAA)	1	GFAA	921014	DCU/DBH
Spike P (ICP/FLAA)		ICP		
ICLP (ICP/FLAA)		ICLP P		
LCS F (GFAA)	1	GFAA M	744 P.91	DCU/DBH
LCS P (ICP/FLAA)		Leeman's LCS		
LCSP (ICP/FLAA) Na, K, Sb		Na, K, Sb		
LCSF TEST	1		921014	DCU/DBH

Continued on Page

Read and Understood By

DC Upcraft  
Signed

11-12-92  
Date

D L Robb  
Signed

11-17-92  
Date

## **APPENDIX B**

### **Forms**



APPENDIX B-1  
Chain of Custody Form (Blank)

COPIES: White & Yellow copies accompany sample shipment to laboratory.  
Pink copy retained by sampler.

— NORTH PACIFIC DIVISION  
— U.S. CORPS OF ENGINEERS  
— 1491 NW GRAHAM AVE  
— TROUTDALE, OR 97060-9503  
— PHONE (503) 665-4166

PROJ. NO.		PROJECT NAME		NO. OF CONTAINERS		Pg 1 of 1																									
SAMPLERS: (Signature)						REMARKS																									
SAMPLE ID #	DATE	STATION LOCATION				8015 MOD	8100 MOD	8010	8260	8000 P/B	8140	8150	9310 (HNO <sub>3</sub> )	9310 (HNO <sub>3</sub> )	8260 P/B																
92ER0381WA	7-23	Kinsate	*12	1	1	2	2	1	1	1	1	1	1	1	1	*NO HNO <sub>3</sub> for Kinsate															
92ER0384WA	7-23	AK-1	10	-	-	2	2	1	1	1	1	1	1	1	1																
92ER0385WA	7-23	Trip Blank	4	-	-	2	2	-	-	-	-	-	-	-	-																

Relinquished by: (Signature)

*[Signature]*

Relinquished by: (Signature)

*[Signature]*

Relinquished by: (Signature)

*[Signature]*

Date / Time

7-23-92 1530

Date / Time

7-23-92 1530

Date / Time

7-23-92 1530

Received by: (Signature)

*[Signature]*

Received by: (Signature)

*[Signature]*

Received for Laboratory by: (Signature)

*[Signature]*

Relinquished by: (Signature)

*[Signature]*

Relinquished by: (Signature)

*[Signature]*

Date / Time

7/23/92 0930

Date / Time

7/27/92 1130

Date / Time

7/27/92 1130

Remarks

7 day turnaround Required

Received by: (Signature)

*[Signature]*

Received by: (Signature)

*[Signature]*

Distribution: Original Accompanies Shipment; Copy to Coordinator Flight Files

Note: Please return signed and dated original chain-of-custody record with data results. Note broken custody seals, leaking or broken bottles.

\* Nn 9310 or 9320 containers received. 6/15/10

APPENDIX B-2  
Chain of Custody Exemplar

SDG#

SAS#

Case

# 9174

Received: 07-28-92

0930

Date: 08-04-92

ARDL # CUST. #

9174-1 92ER0681 WA

WATER

VOC-8260

8015 Mod.

8100 Mod-EPH

HVO-8010

Pest/PCB-8080

8140

8150

Metals (8 RCRA)

Gross Alpha -9310

Rad. um-9320

Collected

2

84WA

3

88WA

7/23

## APPENDIX B-3

Analysis Requirement Flow Sheet

## APPENDIX B-4

### WATER QUALITY

If these criteria are not met corrective action must be taken

**Specific Conductance <1.0**

[illegible]

FORM QA 1

### HOOD VELOCITY and VOLUME

Month-\_\_\_\_\_

[illegible]

Comments:

Signature-\_\_\_\_\_

## WEEKLY BALANCE CHECK

CLASS S WEIGHTS

DATE- \_\_\_\_\_

WEIGHTS	5.0 mg	100.0 mg	500.0 mg	5.0 g	50.0 g	100.0 g
BALANCES						
AF - 1						
AF - 2						
AF - 3						
AF - 4						
AF - 5						
AF - 6						
AF - 7						
AM - 1						
OHAUS						

COMMENTS or CORRECTIVE ACTIONS:

SIGNATURE- \_\_\_\_\_

## MONTHLY BALANCE CHECK

CLASS S WEIGHTS

DATE- \_\_\_\_\_

WEIGHTS	1.0 mg	5.0 mg	20.0 mg	100.0 mg	500.0 mg	5.0 g	50.0 g	100.0 g
BALANCES								
AF - 1								
AF - 2								
AF - 3								
AF - 4								
AF - 5								
AF - 6								
AF - 7								
AM - 1								
OHAUS								

COMMENTS or CORRECTIVE ACTIONS:

SIGNATURE- \_\_\_\_\_

## WAVELENGTH VERIFICATION

MONTH-\_\_\_\_\_

Hitachi UV/VIS

Known Standard Wavelength (nm)	Wavelength at Maximum Absorbance (nm)	Accuracy (nm)

Comments:

Milton-Roy Spec 21

Known Standard Wavelength (nm)	Wavelength at Maximum Absorbance(nm)	Accuracy (nm)

Comments:

Signature-\_\_\_\_\_





### REFRIGERATOR TEMPERATURES

4.0°C +/- 2.0°C

[illegible]

**Legend for Corrective Action:**

↑ Turned up to increase temperature

↓ Turned down to decrease temperature

**Comment column contains any other coorective actions.**

FORM QA 8A

## OVEN / INCUBATOR TEMPERATURES

## 2.0 C +/- from Temperature Setting

[illegible]

FORM QA 8B

## AUTOCLAVE MONITORING

[illegible]

FORM QA 9

## MONTHLY FIRE/SAFTY CHECK SHEET

Month-\_\_\_\_\_

Location:	Fire Ext	Fire blkt	Eye wash	1st Aide	Comments
<b>Admin. Traller</b>					
Copier					
Back Hall off #5					
<b>GC/MS:</b>					
Main Entrance					
Room #1					
Room #2					
Room #3					
Closet					
Bathroom					
<b>GC:</b>					
Main Entrance					
Room #1					
Room #2					
Room #3					
Back Entrance					
Bathroom					
<b>Building #4</b>					
Work Load Sink					
Digestion Lab					
Jarrell Ash Room					
Enviromental Room					
Data Reduction					
Glassware Sink					
AA Lab-2					
Sample Coolers					
<b>Shell Building:</b>					
Entrance					
BOD Incubator					
Small Room					
Glassware Sink					
West Wall Hoods					
Extraction Lab Ent.					
Extraction Lab Hood					
<b>Field Services:</b>					
East End					
West End					

Corrective Action:

SIGNATURE-\_\_\_\_\_

## **APPENDIX C**

**Sample Holding Time, Preservatives,  
Containers and Required Volumes**

## APPENDIX C

TABLE 1

### REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR ARDL WATER-WASTEWATER SAMPLES

<u>Analyte</u>	<u>Container<sup>(1)</sup></u>	<u>Volume Required</u>	<u>Preservative<sup>(2)</sup></u>	<u>Time<sup>(5)</sup></u>
Cyanide, total	P, G	500 ml	Cool, 4°C NaOH to pH 10	12 days
Mercury	G	500 ml	HNO <sub>3</sub> to pH 2	28 days
All Other Metals	P, G	1 L	HNO <sub>3</sub> to pH 2 <sup>(3)</sup>	6 months
Volatiles	G, 40 ml, Teflon lined Septum	80 ml	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(4)</sup>	14 days
Acid-Base Neutrals	G, 4 L, Teflon- lined cap	4 L	Cool, 4°C Store in dark until extraction	7 days to extraction 40 days to analyze
PCB's/Pesticides	G, 4L, Teflon- lined cap	4 L	Cool, 4°C pH 5-9	7 days to extraction 40 days to to analyze

(1) Polyethylene (P) or Glass (G), 1 liter unless otherwise specified.

(2) Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection.

(3) Samples should be filtered immediately on-site before adding preservative for dissolved metals.

(4) Should only be used in the presence of residual chlorine.

(5) Calculated from date of collection or as specified by prior agreement with customer.

APPENDIX C  
TABLE 2

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND  
MAXIMUM HOLDING TIMES FOR ARDL SOIL SAMPLES

Inorganic Tests

Container - 8 to 9 ounce, widemouth glass jar with Teflon lined screwcap.

Preservation - Cool, 4°C, protect from light.

Holding Time<sup>(1)</sup> Before Analysis - 6 months for all metals except:

Mercury - 28 days. Cyanide - 12 days. Total solids - 5 days.

Organic Tests

Volatiles

Container - 9 oz. wide-mouth glass bottle with Teflon lined cap.

Preservation - Cool, 4°C, protect from light.

Holding Time<sup>(1)</sup> Before Analysis - 14 days

Acid-Base Neutrals

Container - 8 to 9 ounce, widemouth glass jar with Teflon lined screwcap.

Preservation - Cool, 4°C, protect from light.

Holding Time (1) Before Extraction - 14 days

Holding Time (2) Before Analysis - 40 days

Pesticide/PCB's

Container - 8 to 9 ounce, widemouth glass jar with Teflon lined screwcap.

Preservation - Cool, 4°C, protect from light.

Holding Time (1) Before Extraction - 14 days

Holding Time (2) Before Analysis - 40 days

(1) - Calculated from date of collection or as specified by prior agreement with customer.

(2) - Calculated from date of sample extraction.



## **APPENDIX D**

### **GC/MS Requirements**

## APPENDIX D.1

### GC/MS TUNING VERIFICATION LIMITS

#### BFB Tuning Verification Limits

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (see note)
173	less than 2.00 percent of mass 174
174	greater than 50.0 to 120 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent of m/z 95.

#### DFTPP Tuning Verification Limits

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30.0 - 80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	Present
70	less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 0.75 percent of mass 198
441	present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

NOTE: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 110 percent of m/z 198.

## APPENDIX D.2

### Target Compounds List - Volatiles

<u>Compounds</u>	<u>Contract Required Quantitation Limits</u>			
	<u>Water</u> (ug/l)	<u>Low</u> <u>Soil</u> (ug/kg)	<u>Med.</u> <u>Soil</u> (ug/kg)	<u>On</u> <u>Column</u> (ng)
chloromethane	10	10	1200	(50)
bromomethane	10	10	1200	(50)
vinyl chloride	10	10	1200	(50)
chloroethane	10	10	1200	(50)
methylene chloride	10	10	1200	(50)
acetone	10	10	1200	(50)
carbon disulfide	10	10	1200	(50)
1,1-dichloroethene	10	10	1200	(50)
1,1-dichloroethane	10	10	1200	(50)
1,2-dichloroethene (total)	10	10	1200	(50)
chloroform	10	10	1200	(50)
1,2-dichloroethane	10	10	1200	(50)
2-butanone	10	10	1200	(50)
1,1,1-trichloroethane	10	10	1200	(50)
carbon tetrachloride	10	10	1200	(50)
dichlorobromomethane	10	10	1200	(50)
1,2-dichloropropane	10	10	1200	(50)
c-1,3-dichloropropene	10	10	1200	(50)
trichloroethene	10	10	1200	(50)
chlorodibromomethane	10	10	1200	(50)
1,1,2-trichloroethane	10	10	1200	(50)
benzene	10	10	1200	(50)
t-1,3-dichloropropene	10	10	1200	(50)
bromoform	10	10	1200	(50)
4-methyl-2-pentanone	10	10	1200	(50)
2-hexanone	10	10	1200	(50)
tetrachloroethene	10	10	1200	(50)
toluene	10	10	1200	(50)
1,1,2,2-tetrachloroethane	10	10	1200	(50)
chlorobenzene	10	10	1200	(50)
ethylbenzene	10	10	1200	(50)
styrene	10	10	1200	(50)
total xylenes	10	10	1200	(50)

NOTE: Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required, will be higher.

### APPENDIX D.3

#### Relative Response Factor Criteria For Initial and Continuing Calibration

##### Volatiles

<u>Volatile Compounds</u>	<u>Minimum</u> <u>RF</u>	<u>Maximum</u> <u>%RSD</u>	<u>Maximum</u> <u>%D</u>
Bromomethane	0.100	20.5	25.0
Vinyl Chloride	0.100	20.5	25.0
1,1-Dichloroethene	0.100	20.5	25.0
1,1-Dichloroethane	0.200	20.5	25.0
Chloroform	0.200	20.5	25.0
1,2-Dichloroethane	0.100	20.5	25.0
1,1,1-Trichloroethane	0.100	20.5	25.0
Carbon Tetrachloride	0.100	20.5	25.0
Bromodichloromethane	0.200	20.5	25.0
cis-1,3-Dichloropropene	0.200	20.5	25.0
Trichloroethene	0.300	20.5	25.0
Dibromochloromethane	0.100	20.5	25.0
1,1,2-Trichloroethane	0.100	20.5	25.0
Benzene	0.500	20.5	25.0
trans-1,3-Dichloropropene	0.100	20.5	25.0
Bromoform	0.100	20.5	25.0
Tetrachloroethene	0.200	20.5	25.0
1,1,2,2-Tetrachloroethane	0.500	20.5	25.0
Toluene	0.400	20.5	25.0
Chlorobenzene	0.500	20.5	25.0
Ethylbenzene	0.100	20.5	25.0
Styrene	0.300	20.5	25.0
Xylene (total)	0.300	20.5	25.0
Bromofluorobenzene	0.200	20.5	25.0

# APPENDIX D.4

## Semi-Volatile Target Compounds

Compounds	Contract Required Quantitation Limits			
	<u>Water</u> (ug/l)	<u>Low</u> <u>Soil</u> (ug/kg)	<u>Med.</u> <u>Soil</u> (ug/kg)	<u>On</u> <u>Column</u> (ng)
Phenol	10	330	10000	(20)
Bis(2-chloroethyl)ether	10	330	10000	(20)
2-Chlorophenol	10	330	10000	(20)
1,3-Dichlorobenzene	10	330	10000	(20)
1,4-Dichlorobenzene	10	330	10000	(20)
1,2-Dichlorobenzene	10	330	10000	(20)
2-Methylphenol	10	330	10000	(20)
2,2'-oxybis(1-Chloropropane)	10	330	10000	(20)
4-Methylphenol	10	330	10000	(20)
N-Nitroso-di-n-propylamine	10	330	10000	(20)
Hexachloroethane	10	330	10000	(20)
Nitrobenzene	10	330	10000	(20)
Isophorone	10	330	10000	(20)
2-Nitrophenol	10	330	10000	(20)
2,4-Dimethylphenol	10	330	10000	(20)
bis(2-Chloroethoxy)methane	10	330	10000	(20)
2,4-Dichlorophenol	10	330	10000	(20)
1,2,4-Trichlorobenzene	10	330	10000	(20)
Naphthalene	10	330	10000	(20)
4-Chloroaniline	10	330	10000	(20)
Hexachlorobutadiene	10	330	10000	(20)
4-Chloro-3-methylphenol	10	330	10000	(20)
2-Methylnaphthalene	10	330	10000	(20)
Hexachlorocyclopentadiene	10	330	10000	(20)
2,4,6-Trichlorophenol	10	330	10000	(20)
2,4,5-Trichlorophenol	50	800	50000	(100)
2-Chloronaphthalene	10	330	10000	(20)
2-Nitroaniline	50	800	50000	(100)
Dimethylphthalate	10	330	10000	(20)
Acenaphthylene	10	330	10000	(20)
2,6-Dinitrotoluene	10	330	10000	(20)
3-Nitroaniline	50	800	50000	(100)
Acenaphthene	10	330	10000	(20)
2,4-Dinitrophenol	50	800	50000	(100)
4-Nitrophenol	50	800	50000	(100)
Dibenzofuran	10	330	10000	(20)
2,4-Dinitrotoluene	10	330	10000	(20)

**APPENDIX D.4**

(Continued)

**Semi-Volatile Target Compounds**

Compounds	<u>Contract Required Quantitation Limits</u>			
	<u>Water</u> (ug/l)	<u>Low</u> <u>Soil</u> (ug/kg)	<u>Med.</u> <u>Soil</u> (ug/kg)	<u>On</u> <u>Column</u> (ng)
Diethylphthalate	10	330	10000	(20)
4-Chlorophenyl-phenyl ether	10	330	10000	(20)
Fluorene	10	330	10000	(20)
4-Nitroaniline	25	800	25000	(50)
4,6-Dinitro-2-methylphenol	25	800	25000	(50)
N-Nitrosodiphenylamine	10	330	10000	(20)
4-Bromophenyl-phenyl ether	10	330	10000	(20)
Hexachlorobenzene	10	330	10000	(20)
Pentachlorophenol	25	800	25000	(50)
Phanathrene	10	330	10000	(20)
Anthracene	10	330	10000	(20)
Carbazole	10	330	10000	(20)
Di-n-butylphthalate	10	330	10000	(20)
Fluoranthene	10	330	10000	(20)
Pyrene	10	330	10000	(20)
Butyl benzyl phthalate	10	330	10000	(20)
3,3'-Dichlorobenzidene	20	330	10000	(20)
Benzo (a) anthracene	10	330	10000	(20)
Chrysene	10	330	10000	(20)
Bis (2-ethylhexyl) phthalate	10	330	10000	(20)
Di-n-octyl phthalate	10	330	10000	(20)
Benzo (b) fluoranthene	10	330	10000	(20)
Benzo (k) fluoranthene	10	330	10000	(20)
Benzo (a) pyrene	10	330	10000	(20)
Indeno (1,2,3-cd) pyrene	10	330	10000	(20)
Dibenzo (a,h) anthracene	10	330	10000	(20)
Benzo (g,h,i) perylene	10	330	10000	(20)

## APPENDIX D.5

### Relative Response Factor Criteria For Initial and Continuing Calibration

#### Semi-Volatiles

<u>Volatile Compounds</u>	<u>Minimum</u> <u>RF</u>	<u>Maximum</u> <u>%RSD</u>	<u>Maximum</u> <u>%D</u>
Phenol	0.800	20.5	25.0
bis(2-Chloroethyl)ether	0.700	20.5	25.0
2-Chlorophenol	0.800	20.5	25.0
1,3-Dichlorobenzene	0.600	20.5	25.0
1,4-Dichlorobenzene	0.500	20.5	25.0
1,2-Dichlorobenzene	0.400	20.5	25.0
2-Methylphenol	0.700	20.5	25.0
4-Methylphenol	0.600	20.5	25.0
N-Nitroso-di-n-propylamine	0.500	20.5	25.0
Hexachloroethane	0.300	20.5	25.0
Nitrobenzene	0.200	20.5	25.0
Isophorone	0.400	20.5	25.0
2-Nitrophenol	0.100	20.5	25.0
2,4-Dimethylphenol	0.200	20.5	25.0
bis(2-Chloroethoxy)methane	0.300	20.5	25.0
2,4-Dichlorophenol	0.200	20.5	25.0
1,2,4-Trichlorobenzene	0.200	20.5	25.0
Naphthalene	0.700	20.5	25.0
4-Chloro-3-methylphenol	0.200	20.5	25.0
2-Methylnaphthalene	0.400	20.5	25.0
2,4,6-Trichlorophenol	0.200	20.5	25.0
2,4,5-Trichlorophenol	0.200	20.5	25.0
2-Chloronaphthalene	0.800	20.5	25.0
Acenaphthylene	1.300	20.5	25.0
2,6-Dinitrotoluene	0.200	20.5	25.0
Acenaphthene	0.800	20.5	25.0
Dibenzofuran	0.800	20.5	25.0
2,4-Dinitrotoluene	0.200	20.5	25.0
4-Chlorophenyl-phenylether	0.400	20.5	25.0
Fluorene	0.900	20.5	25.0
4-Bromophenyl-phenylether	0.100	20.5	25.0
Hexachlorobenzene	0.100	20.5	25.0
Pentachlorophenol	0.050	20.5	25.0
Phenanthrene	0.700	20.5	25.0
Anthracene	0.700	20.5	25.0
Fluoranthene	0.600	20.5	25.0

**APPENDIX D.5**

**(Continued)**

**Relative Response Factor Criteria**  
**For Initial and Continuing Calibration**

**Semi-Volatiles**

<b><u>Volatile Compounds</u></b>	<b><u>Minimum RF</u></b>	<b><u>Maximum %RSD</u></b>	<b><u>Maximum %D</u></b>
Pyrene	0.600	20.5	25.0
Benzo(a)anthracene	0.800	20.5	25.0
Chrysene	0.700	20.5	25.0
Benzo(b)fluoranthene	0.700	20.5	25.0
Benzo(k)fluoranthene	0.700	20.5	25.0
Benzo(a)pyrene	0.700	20.5	25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	25.0
Dibenz(a,h)anthracene	0.400	20.5	25.0
Benzo(g,h,i)perylene	0.500	20.5	25.0
Nitrobenzene-d5	0.200	20.5	25.0
2-Fluorobiphenyl	0.700	20.5	25.0
Terphenyl-d14	0.500	20.5	25.0
Phenol-d6	0.800	20.5	25.0
2-Fluorophenol	0.600	20.5	25.0
2-Chlorophenol d4	0.800	20.5	25.0
1,2-Dichlorobenzene d4	0.400	20.5	25.0

Target Compounds not listed in Table 3 do not have %RSD or %D criteria. However, Target Compounds and surrogates not listed in Table 3 must meet a minimum RF criterion of 0.010.



APPENDIX D.6

VOLATILE/SEMIVOLATILE

Surrogate Compound Percent Recovery Criteria

<u>Fraction</u>	<u>Surrogate Compound</u>	<u>Low/Medium Water</u>	<u>Low/Medium Soil/Sediment</u>
VOA	Toluene-d8	88-110	87-138
VOA	4-Bromofluorobenzene	86-115	59-113
VOA	1,2-Dichloroethane-d4	76-114	70-121
SV	Nitrobenzene-d5	35-114	23-120
SV	2-Fluorobiphenyl	43-116	30-115
SV	p-Terphenyl-d4	33-141	18-137
SV	Phenol-d5	10-110	24-113
SV	2-Fluorophenol	21-110	25-121
SV	2,4,6-Tribromophenol	10-123	19-122
SV	2-Chlorophenol-d4	33-110*	20-130*
SV	1,2-Dichlorobenzene-d4	16-110*	20-130*

\* At present, these recovery limits are advisory only. They are not used to determine if a sample should be reanalyzed.

APPENDIX D.7

MATRIX SPIKE DUPLICATE QC LIMITS

Volatile/Semi-volatile

<u>Fraction</u>	<u>Matrix Spike Compound</u>	<u>Recovery Limits</u>		<u>RPD Limits</u>	
		<u>Water</u>	<u>Soil/Sed</u>	<u>Water</u>	<u>Soil/Sed</u>
VOA	1,1-Dichloroethene	61-145	59-172	14	22
VOA	Trichloroethene	71-120	62-137	14	24
VOA	Benzene	76-127	66-142	11	21
VOA	Toluene	76-125	59-139	13	21
VOA	Chlorobenzene	75-130	60-133	13	21
SV	Phenol	12-110	26-90	42	35
SV	2-Chlorophenol	27-123	25-102	40	50
SV	1,4-Dichlorobenzene	36-97	28-104	28	27
SV	N-nitroso-di-n-propylamine	41-116	41-125	38	38
SV	1,2,4-Trichlorobenzene	39-98	38-107	28	23
SV	4-Chloro-3-methylphenol	23-97	26-103	42	33
SV	Acenaphthene	46-118	31-137	31	19
SV	4-Nitrophenol	10-80	11-114	50	50
SV	2,4-Dinitrotoluene	24-96	28-89	38	47
SV	Pentachlorophenol	9-103	17-109	50	47
SV	Pyrene	26-127	35-142	31	36

## **APPENDIX E**

### **Gas Chromatography Requirements**

## APPENDIX E.1

### Pesticide Target Compounds

<u>Compounds</u>	<u>Contract Required Quantitation Limits</u>	
	<u>Water</u> (ug/l)	<u>Low Soil</u> (ug/kg)
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
Lindane (gamma-BHC)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3
4,4'-DDT	0.10	3.3
Methoxychlor	0.50	17.0
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.05	1.7
gamma-Chlordane	0.05	1.7
Toxaphene	5.0	170.0
Arochlor-1016	1.0	33.0
Arochlor-1221	2.0	67.0
Arochlor-1232	1.0	33.0
Arochlor-1242	1.0	33.0
Arochlor-1248	1.0	33.0
Arochlor-1254	1.0	33.0
Arochlor-1260	1.0	33.0

NOTE: There is no differentiation between the preparation of low and medium soil samples for the analysis of Pesticides/Arochlors.

APPENDIX E.2  
METHOD DETECTION LIMITS FOR VOLATILE ORGANICS

<u>Compound</u>	<u>Method Detection Limit<sup>a</sup></u> (ug/L)
Benzyl chloride <sup>b</sup>	
Bis(2-chloroethoxy)methane <sup>c</sup>	
Bromobenzene	
Bromodichloromethane	0.10
Bromoform	0.20
Bromomethane	
Carbon tetrachloride	0.12
Chlorobenzene	0.25
Chloroethane	0.52
2-Chloroethyl vinyl ether	0.13
Chloroform	0.05
1-Chlorohexane	
Chloromethane	0.08
Chloromethylmethyl ether <sup>c</sup>	
Chlorotoluene <sup>b</sup>	
Dibromochloromethane	0.09
Dibromomethane	
1,2-Dichlorobenzene	0.15
1,3-Dichlorobenzene	0.32
1,4-Dichlorobenzene	0.24
Dichlorodifluoromethane <sup>b</sup>	
1,1-Dichloroethane	0.07
1,2-Dichloroethane	0.03
1,1-Dichloroethylene	0.13
trans-1,2-Dichloroethylene	0.10
Dichloromethane	
1,2-Dichloropropane	0.04
trans-1,3-Dichloropropylene	0.34
1,1,2,2-Tetrachloroethane	0.03
1,1,1,2-Tetrachloroethane	
Tetrachloroethylene	0.03
1,1,1-Trichloroethane	0.03
1,1,2-Trichloroethane	0.02
Trichloroethylene	0.12
Trichlorofluoromethane	
Trichloropropane	
Vinyl chloride	0.18
Benzene	0.2
Chlorobenzene	0.2
1,4-Dichlorobenzene	0.3
1,3-Dichlorobenzene	0.4
1,2-Dichlorobenzene	0.4
Ethyl Benzene	0.2
Toluene	0.2
Xylenes	

<sup>a</sup>Using purge-and-trap method (Method 5030). Limit may vary dependent on sample matrix.

<sup>b</sup>Demonstrated very erratic results when tested by purge-and-trap.

<sup>c</sup>Demonstrated poor purging efficiency.

APPENDIX E.3

TARGET ANALYTES AND DETECTION LIMITS  
FOR CHLORINATED HERBICIDES

<u>Compound</u>	<u>Method Detection Limit<sup>(1)</sup></u> (ug/L)
2,4-D	1.2
2,4-DB	0.91
2,4,5-T	0.20
2,4,5-TP (Silvex)	0.17
Dalapon	5.8
Dicamba	0.27
Dichloroprop	0.65
Dinoseb	0.07
MCPA	249
MCP	192

(1) Limit may vary, dependent on sample matrix.

#### APPENDIX E.4

### TARGET ANALYTES AND DETECTION LIMITS FOR ORGANOPHOSPHORUS PESTICIDES

<u>Compound</u>	<u>Method Detection Limit<sup>(1)</sup></u> (ug/L)
Azinphos methyl	1.5
Bolstar	0.15
Chlorpyrifos	0.3
Coumaphos	1.5
Demeton-O	0.25
Demeton-S	0.25
Diasinon	0.6
Dichlorvos	0.1
Disulfoton	0.20
Ethoprop	0.25
Fensulfothion	1.5
Fenthion	0.10
Merphos	0.25
Mevinphos	0.3
Naled	0.1
Parathion methyl	0.03
Phorate	0.15
Ronnel	0.3
Stiropfos (Tetrachlorvinphos)	5.0
Tokuthion (Prothiofos)	0.5
Trichloronate	0.15

(1) Limits may vary, dependent on sample matrix.

APPENDIX E.5

CHLORINATED PESTICIDE/PCB SPIKE AND SURROGATE LIMITS

<u>Matrix Spike Compound</u>	<u>Recovery Limity</u>		<u>RPD Limits</u>	
	<u>Water</u>	<u>Soil/Sed</u>	<u>Water</u>	<u>Soil/Sed</u>
Lindane	56-123	46-127	15	50
Heptachlor	40-131	35-130	20	31
Aldrin	40-120	34-132	22	43
Dieldrin	52-126	31-134	18	38
Endrin	56-121	42-139	21	45
Arochlor 1254	(1)	(1)	30	50
 <u>Surrogate Compound</u>				
Tetrachloro-m-xylene	60-150	60-150		
Decachlorobiphenyl	60-150	60-150		

(1) No values available.



## **APPENDIX F**

### **Inorganic Analysis Requirements**

APPENDIX F.1

INORGANIC TARGET ANALYTES

<u>Metals</u>	<u>(CRDL)-ug/l</u>
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20

## APPENDIX F.2

### SPIKING LEVELS FOR INORGANIC ANALYSIS<sup>(1)</sup>

<u>Element</u>	<u>For ICP/Flame AA</u>		<u>For Furnace AA</u>	
	<u>(ug/L)</u>		<u>(ug/L)</u>	
	<u>Water<sup>(2)</sup></u>	<u>Sediment<sup>(3)(4)</sup></u>	<u>Water</u>	<u>Sediment</u>
Aluminum	2,000	*		
Antimony	500	500	100	100
Arsenic	2,000	2,000	40	40
Barium	2,000	2,000		
Beryllium	50	50		
Cadmium	50	50	5	5
Calcium	*	*	*	*
Chromium	200	200		
Cobalt	500	500		
Copper	250	250		
Iron	1,000	*		
Lead	500	500	20	20
Magnesium	*	*	*	*
Manganese	500	500		
Mercury			1	1
Nickel	500	500		
Potassium	*	*	*	*
Selenium	2,000	2,000	10	10
Silver	50	50		
Sodium	*	*	*	*
Thallium	2,000	2,000	50	50
Vanadium	500	500		
Zinc	500	500		
Cyanide <sup>(5)</sup>				

- (1) Amount to add prior to digestion/distillation -- choose amount appropriate to method of analysis. Elements without spike levels and not designated with an asterisk, should be spiked at appropriate levels.
  - (2) Sample matrices of ground, surface or wastewater.
  - (3) The levels shown indicate concentrations in the digestate of the spiked sample. See text of Section 7.1.3 for discussion.
  - (4) Sample matrices of solid, soil, sediment or sludge.
  - (5) For waters add 100 ug/l to original sample volume. For soils, appropriate spikes are added as indicated in text of Section 7.1.3.
- \* No spike required.

## **APPENDIX G**

### **Analytical Methods**

APPENDIX G.1

METHODS FOR TRACE METAL ANALYSIS<sup>(1)</sup>

<u>Analyte</u>	<u>SW 846</u>	<u>40 CFR 136</u>	<u>40 CFR 141</u>
Aluminum	6010	200.7	-
Antimony	6010	200.7	-
Arsenic	6010/7061	206.3	206.3
Barium	6010	200.7	200.7
Beryllium	6010	200.7	-
Boron	6010	200.7	-
Cadmium	6010	200.7	200.7
Calcium	6010	200.7	200.7
Chromium	6010	200.7	200.7
Cobalt	6010	200.7	-
Copper	6010	200.7	200.7
Iron	6010	200.7	200.7
Lead	6010/7421	200.7/239.2	239.2
Magnesium	6010	200.7	-
Manganese	6010	200.7	200.7
Mercury <sup>(2)</sup>	7470/7471	245	245
Nickel	6010	200.7	-
Potassium	6010	200.7	-
Selenium	6010/7741	270.3	270.2
Silver	6010	200.7	200.7
Sodium	6010	200.7	200.7
Thallium	6010/7841	200.7/279.2	-
Vanadium	6010	200.7	-
Zinc	6010	200.7	200.7

(1) Where dual methods are cited (except for mercury), the second method will be used as required to reach detection limits appearing in Appendix F.

(2) Methods cited employed as dictated by sample matrix.

APPENDIX G.2

METHODS FOR INORGANIC ANALYSIS AND  
DETERMINATION OF PHYSICAL PROPERTIES<sup>(1)</sup>

<u>Analyte</u>	<u>SW 846</u>	<u>40 CFR 136</u>	<u>40 CFR 141</u>
Acidity	-	305	-
Alkalinity	-	310	310.1
Bromide	-	320.1	-
Biochemical Oxygen Demand	-	405.1	-
Cation Exchange Capacity	9080	-	-
Chemical Oxygen Demand	-	410	-
Chloride	9250/9252	325	300
Chlorine, Residual	-	330.2/330.3	-
Color	-	110.2	-
Conductance	9050	120.1	-
Corrosivity (NACE)	1110	1110	-
Corrosivity (pH)	9040	150.3	-
Cyanide, Amenable	9012	335.1	-
Cyanide, Total	9012	335.3	335.2
Flash Point	1010	-	-
Fluoride	-	340.2	340.2
Hardness	-	130.1	-
Iodide	-	345.1	-
Nitrogen, Ammonia	-	350.1/350.3	-
Nitrogen, Total	-	351.2/351.4	-
Nitrogen, Nitrate-Nitrite	-	353	353/300
Nitrogen, Nitrite	-	354.1	354.1/353.1
Oil and Grease, Total	9070	413.1	-
Organic Carbon, Total	9060	415	-
Organic Halides	9020/9021	-	-
Oxygen, Dissolved	-	360.1	-
Paint Filter Test	9095	-	-
Petroleum Hydrocarbons	9071(2)	418.1	-
pH	9040/9045	150.1/150.2	150.1/150.2
Phenolics	9066/9065	420.2	-
Phosphorus, Total	-	365.2/365.4	-
Reactivity	Chapter 7	-	-
Residue, Filterable	-	160.1	160.1
Residue, Non-Filterable	-	160.2	-
Residue, Total	-	160.3	-
Residue, Volatile	-	160.4	-
Settleable Matter	-	160.5	-
Silicon, Dissolved	-	370.1	-

APPENDIX G.2  
(continued)

METHODS FOR INORGANIC ANALYSIS AND  
DETERMINATION OF PHYSICAL PROPERTIES<sup>(1)</sup>

<u>Analyte</u>	<u>SW 846</u>	<u>40 CFR 136</u>	<u>40 CFR 141</u>
Sulfate	9038	375.4	375.4/300
Sulfide	9030/9031	376.1	-
Surfactants (MBAS)	-	425.1	-
Temperature	-	170.1	-
Turbidity	-	180.1	-

- (1) Dual citation of methods involves two sample matrices or use of second method to meet detection limits in Appendix F.
- (2) Extraction method. Quantitation by 418.1.

### APPENDIX G.3

#### METHODS FOR ORGANICS ANALYSIS<sup>(1)</sup>

<u>Analyte</u>	<u>SW 846</u>	<u>40 CFR 136</u>	<u>40 CFR 141</u>
Herbicides, Acidic <sup>(2)</sup>	-	-	515
Herbicides, Chlorinated <sup>(2)</sup>	8150	509B <sup>(3)</sup>	-
PCB's	8080	608	-
Pesticides, Carbamates	-	-	531
Pesticides, Halogenated	8080	608	508
Pesticides, Phosphorus	8051	-	507
Phenols	8040	-	-
Phthalate Esters	8060	606	-
Polynuclear Hydrocarbons	8100	610	-
Semivolatiles	8270	625	-
Trihalomethanes	-	-	501.1
Volatiles (GC/MS)	8240/8260	624	524.1/524.2
Volatile Aromatics	8020	602	503.1
Volatile Halocarbons	8010	601	502.1
Polynuclear Aromatic Hydrocarbons	8310	-	-

- (1) Dual citation of methods involves two sample matrices or use of second method to meet detection limits in Appendix F.
- (2) Derivatization accomplished using boron trifluoride rather than azomethane.
- (3) Reference 3, Introduction.



APPENDIX G.4

METHODS FOR RADIOACTIVITY ANALYSIS

<u>Analyte</u>	<u>SW 846</u>	<u>Standard Methods<sup>(1)</sup></u>	<u>40 CFR 141</u>
Cesium-134/137	-	750-Cs	901.0
Gross alpha, gross beta	9310	7110	900.0
Iodine-131	-	7500-I	902.0
Radium, alpha emitters	9315	7500-RaB	901.1
Radium 226 (Radon emanation)	-	7500-RaC	903.0
Radium-228	9320	7500-RaB	904.0
Strontium-90	-	7500-Sr	905.0
Tritium	-	7500-H	906.0

(1) Reference 3, Introduction.

## **APPENDIX H**

### **Preventative Maintenance Schedules**

## APPENDIX H.1

### Balance Preventative Maintenance

#### **1.0**      Overall Maintenance

Aside from routine calibration checks and cleaning external surfaces, no maintenance is performed on any balances in the laboratory. All analytical balances are covered by a maintenance agreement which involves annual inspection, service and calibration by a qualified technician and provides for emergency service calls as required. Top loading balances are not covered by such an agreement. If one of these instruments fails to satisfy criteria during a routine calibration check or performs unsatisfactorily in some other way, it is returned to the manufacturer for service.

#### **2.0**      Calibration Checks

A Denver Instruments Analytical Balance Model AB-250D, Serial Number B042397 is designated the primary balance. The instrument is mounted on a slate table which is separated from all counters and cabinets. The instrument is located in an area where traffic is minimum and the effects of drafts and temperature and humidity fluctuations are controlled.

This balance (and all other analytical balances at ARDL) is cleaned and calibrated annually by a factory authorized technician. Class S weights traceable to the National Bureau of Standards are used in the calibration and a certificate of calibration is retained on file.

On a monthly basis the calibration of the Denver is checked using a set of Class S weights. Once that calibration has been verified, the same weights are used to check calibration on the other balances in use in the laboratory. All data are recorded on Form QA-3 and the precision of the readings is calculated based on the true values obtained with the Denver. If the values observed on any balance are not within 98-102% of true, that instrument is removed from service until the problem is defined and corrected. All maintenance on balances (except for external cleaning) is performed by factory authorized technicians.

Before beginning daily use, each balance is checked for accuracy with a single Class S weight. The observed and true values for the weight are recorded in the logbook in use by the analyst. If the observed value for the weight is not within 98-102% of the true value, the instrument is removed from service until the problem is defined and corrected.

#### **3.0**      Daily Use

Prior to use, the level of the balance is checked. When level is satisfactory the balance is zeroed and the accuracy is checked using a single Class S weight as described above.

When work is completed on analytical balances, all weights are removed and power to the instrument is turned off. After cleaning the pan and the weighing compartment, the doors to the compartment are closed and the balance left in standby condition, ready for next use.

When work is completed on top loading balances, power is turned off and the pan and top surfaces cleaned. The balance is left in standby, ready for next use.

APPENDIX B.4.1

BALANCE CALIBRATION

	Lab 1		Lab
3	Sartorius	Mettler	
Ainsworth	Analytical	Top Loader	Top
Loader	Model 2432	Model PE300	Model
300-DR	Serial # 132318	Serial # D79063	Serial
# 86-6497			
	<u>Weight(g)</u>	<u>Weight(g)</u>	<u>Difference(%)</u>
<u>Weight(g)</u>	<u>Difference(%)</u>		
10 mg			
20			
50			
100			
200			
500			
1 g			
2			
3			
5			
10			
20			
30			
50			
100			
150			
190			

BALANCE CALIBRATION

Bldg. 4 Hood Mettler Analytical AC100 Serial # A56580 <u>Weight(g) Difference(%)</u>	Bldg. 4 Balance Table Mettler Analytical AC100 Serial # A88839 <u>Weight(g) Difference(%)</u>	O'Haus Environmental Room Brain Weigh B 300 DO Serial # 11279 <u>Weight(g) Difference(%)</u>
---	--	--

10 mg

20

50

100

200

500

1 g

2

3

5

10

20

30

50

100

150

190

Form QA-3

\_\_\_\_\_  
Date

\_\_\_\_\_  
Analyst

## APPENDIX H.2

### Preventative Maintenance, pH and Specific Ion Meters and Electrodes

#### 1.0 Logbook

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

#### 2.0 Meter

Before use, battery power is checked by manipulation of front panel controls in accordance with instructions in the operating manual for the meter. If the front panel indication is not satisfactory, the meter is removed from service and the batteries replaced.

After the battery check is complete, the meter is calibrated in accordance with instructions in the operating manual following the procedures outlined in Section 5.1, above. If the meter fails to respond appropriately it is removed from service and appropriate corrective action taken to restore proper function. In general this corrective action usually involves work with the electrode as noted in Section 2.0, below. If the electrode is functioning properly the meter is returned to the manufacturer for repair.

When sample measurements are complete, power to the unit is turned off and all other controls returned to their startup positions. The meter face and upper surfaces are wiped clean, the electrode is stored as described below and the unit is left, ready for next use.

#### 3.0 Electrodes

##### 3.1 Storage

Storage requirements for electrodes not in use vary according to the anticipated duration of the storage period, electrode type and junction construction. The specific instructions for storage furnished by the manufacturer for each electrode are followed routinely.

For short term storage, these instructions usually involve keeping the electrode immersed in a specific solution. Fluoride electrodes, however, are stored in air after rinsing with reagent water and blotting dry. Electrodes for pH are immersed in pH 7 buffer solution containing 0.5% (w/v) potassium chloride. Ammonia electrodes are stored immersed in saturated ammonium chloride.

For long term storage all electrodes are capped and the filling vents are securely closed. Fluoride electrodes are stored in air but for all other electrodes a few drops of the appropriate solutions noted above are placed in the tip cover before it is fitted in place. When

withdrawing electrodes from long term storage the internal electrolyte is replaced with fresh solution and the electrode is immersed in an appropriate solution for at least twenty four hours before use.

### 3.2 Improper Instrument Operation

An electrode fault is usually the cause of improper instrument function. The electrode may be dirty, scratched, cracked, improperly filled or otherwise impaired in operation. By following the routine procedures outlined below, electrode faults can be minimized.

#### 3.2.1 Inspection

At least weekly, inspect electrodes for scratches, cracks, nicks or other physical damage. Also inspect for salt crystal buildup around the membrane/junction interface and for the presence of a coating or film on the electrode tip.

Discard physically damaged electrodes and replace with new ones. If salt buildup has occurred or the electrode is dirty follow the cleaning procedures noted below.

#### 3.2.2 Cleaning

If salt buildup has occurred, rinse the electrode thoroughly in reagent water, empty the internal electrolyte and replace with fresh solution. Immerse the electrode in storage solution for at least twenty four hours before use.

Soaking a dirty electrode in M/10 hydrochloric or nitric acid for one half hour will clean most deposits from the electrode surfaces. Soaking in acid should be followed by at least one hour immersion in storage solution before attempting calibration.

Protein deposits are removed by soaking for 15 minutes in a 1% (w/v) solution of pepsin prepared from M/10 hydrochloric acid. Inorganic deposits are removed by soaking for 15 minutes in M/10 tetrasodium EDTA. Greased and oil are removed by rinsing with mild detergent or a 75% (v/v) aqueous methanol solution.

After performing any of these cleaning procedures, the junction should be rinsed thoroughly in reagent water and the electrode soak in storage solution for at least one hour before calibration or other use is attempted.



## APPENDIX H.3

### Preventative Maintenance, Dissolved Oxygen Meter

#### 1.0 Logbook

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

#### 2.0 Daily

Manipulate the front panel controls on the instrument as described in the operating manual to check batteries, zero and full scale. If improper readings are observed in any case, remove the instrument from service and take appropriate corrective action.

Inspect the electrode membrane carefully. The membrane must be clean and free from surface imperfections and there should be no bubbles in the internal electrolyte solution. If any problems are noted, replace the membrane and the internal electrolyte according to the instructions in the instrument operating manual.

When work with instrument is completed turn off the power. Store the probe in a clean BOD bottle filled with 300 ml of reagent water.

#### 3.0 Monthly

Change membrane and electrolyte solution according to the instructions given in the operating manual for the instrument.

## APPENDIX H.4

### Preventative Maintenance, Atomic Absorption Spectrophotometers

#### 1.0 Overall Maintenance

Overall maintenance on all atomic absorption spectrophotometers is covered by a maintenance agreement with the manufacturer. That agreement provides all parts and labor necessary to keep the instruments in operating condition. Aside from the routine procedures described below, all service and maintenance of this equipment is performed by qualified technicians employed by the instrument manufacturer.

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

Annually, arrange for a preventative maintenance call from a manufacturer's representative.

If a properly maintained instrument fails to respond properly to startup routines or will not meet specifications when in operation, inform the supervisor and take steps to arrange for a service call.

#### 2.0 Hollow Cathode Lamps

Single element hollow cathode lamps are employed in the laboratory. All lamps are dated when received and again when they are first placed into service. Records are maintained in order to track useful life. When sensitivity is lost and/or background noise develops, the tube is replaced regardless of the time it has been used.

#### 3.0 Instrument Maintenance

Clean the exterior surfaces of the instrument housing and sample dispenser with a soft, damp cloth at the end of each working day. If required, stains may be removed with warm, soapy water. Do not use solvents, solvent-based cleaners or harsh abrasives since these materials can damage the exterior finish.

Before attempting use of any atomic absorption spectrophotometer, inspect the gas supplies which will be used with the unit. Replace cylinders as required to ensure a sufficient reserve to complete the planned analytical runs. Verify that the exhaust vents are operating properly and that ventilation is adequate.

### 3.1 Furnace Unit

#### 3.1.1 Furnace

Before beginning work inspect the interior of the graphite shield, electrodes, tube insert, furnace chimney, atomizer capillary tip and all hoses and fittings for dirt, wear and/or damage. Clean or replace as appropriate, following the instructions given in the operating manual for the instrument.

Inspect the quartz windows for cleanliness. Clean both sides of these windows regularly with alcohol and lens paper. If the windows become scratched, clouded or otherwise defective, replace with new units.

#### 3.1.2 Autosampler

Inspect the autosampler, especially the syringe, syringe tip, rinse bottle and all hoses and fittings. If dirty or damaged components are noted, clean or replace as required following the instructions given in the instrument operating manual.

Examine the sampler syringe and assembly for cleanliness and signs of wear or damage. Replace or clean using the procedures given in the instrument operating manual. If air bubbles are present in the syringe or syringe inlet tubing, remove following the procedures described in the instrument operating manual. Regularly clean the rinse bottle using the procedures described for trace metals glassware in Standard Operating Procedures, Glassware Preparation.

### 3.2 Flame Unit

Empty the sample drain reservoir and ensure that the drain line is completely open. Inspect the nebulizer inlet tubing for blockage. Replace or clear as required. Inspect the nebulizer for signs of wear or deterioration. Rebuild, repair or replace as required. If the nebulizer is disassembled for cleaning or repair, adjust the nebulizer bung and glass bead on reassembly to achieve the level of sensitivity specified in the instrument operator's manual.

Inspect the burner head to ensure it is clean and ready for use. Clean as required if the inspection indicates any problems. Inspect the flashback safety valve to ensure that it is clear and fully functional. Also inspect the burner head. Clean as required. Do not disassemble the burner head under any circumstances since an explosion can result if it is operated after improper reassembly.

At the end of each working day thoroughly flush the nebulizer/spray chamber by aspirating at least 50 ml of reagent water through the system.

## APPENDIX H.5

### Preventative Maintenance, Inductively Coupled Argon Plasma Emission Spectrometers

#### 1.0 Overall Maintenance

Overall maintenance on all plasma emission spectrometers is covered by maintenance agreements with the manufacturers. That agreement provides for preventative maintenance visits and for all parts and labor necessary to keep the instruments operating according to specifications. Aside from the routine procedures described below, all service and maintenance of this equipment is performed by qualified technicians employed by the instrument manufacturer.

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

If a properly maintained instrument fails to respond properly to startup routines or will not meet specifications when in operation, inform the supervisor and take steps to arrange for a service call.

#### 2.0 Simultaneous Instrument

A Jarrel-Ash Model 965 spectrometer powered by a standalone plasmatherm Model 2000D RF generator is currently in use in the laboratory.

#### 2.1 RF Generator

Leave main power to the generator on at all times to prolong vacuum tube life and keep the cooling fan in operation. At least weekly inspect the inlet air filter at the back of the unit and replace as required to maintain unrestricted flow of cooling air.

Occasionally clean the exterior surfaces of the cabinet by wiping with a damp rag.

As required, replace the power amplifier tube (5CX1500A) with a new tube. Under no circumstances use rebuilt tubes for replacements. When performing this procedure, turn off all power to the unit and follow the explicit instructions in the operating manual for the generator. There may be residual LETHAL voltages in the interior of the cabinet, even though the unit has not been operated recently. Before beginning tube replacement, discharge all capacitors to ground using the shorting rod provided. Further, if the generator has been in recent operation, the power amplifier tube may be hot enough to cause burns.

No other maintenance on this unit should be performed unless the manufacturer has been consulted.

At the end of the working day, turn the high voltage control fully counterclockwise, turn off the automatic power control and disable the RF circuitry with the front panel switch.

## 2.2 Spectrometer

### 2.2.1 Sample Introduction System

Before beginning work, inspect the peristaltic pump tubing for signs of wear. Inspect the capillary inlet tubing and sipper tube for cleanliness. Replace with new tubing as required.

Inspect the plasma torch, mixing chamber and nebulizer for cleanliness and for the presence of solids which may impede free flow of sample and/or plasma gas. As required, disassemble and clean or replace following the instructions given in the instrument operating manual. When cleaning is complete, reinstall and align the torch following the procedures given in the instrument operating manual.

While the system is in operation, aspirate a solution of 5% (v/v) nitric acid into the sample introduction system to minimize salt buildup. At least weekly, aspirate 50 to 100 ml of detergent solution (3 to 6 drops Triton X in reagent water) into the sample introduction system to minimize buildup of organic and other films on interior surfaces.

### 2.2.2 Working Coil Cooling Water

Inspect the inlet supply line filter at least weekly. Change the filter cartridge at least monthly or more often, as indicated by the appearance of the filter.

### 2.2.3 Data Files

At least weekly evaluate the space available on the data system hard drive using the on board file management software. As indicated, transfer files to diskettes or tape. Maintain at least 10 megabytes of free space on the hard drive.

## 3.0 Sequential Unit

A Leeman Labs Plasmaspec with an on board RF generator is currently in use in the laboratory. Deionized cooling water for the working coil is provided by a closed recirculator system.

### 3.1 Power Supply

Inspect the inlet air filter. Clean or replace as needed. All other maintenance on this unit is performed by qualified service technicians employed by the manufacturer.

## 3.2 Spectrometer

### 3.2.1 Sample Introduction

Before beginning work, inspect the peristaltic pump tubing for signs of wear. Inspect the capillary inlet tubing and sipper tube for cleanliness. Replace with new tubing as required.

Inspect the plasma torch, mixing chamber and nebulizer for cleanliness and for the presence of solids which may impede free flow of sample and/or plasma gas. As required, disassemble and clean or replace following the instructions given in the instrument operating manual. When cleaning is complete, reinstall and align the torch following the procedures given in the instrument operating manual.

While the system is in operation, aspirate a solution of 5% (v/v) nitric acid into the sample introduction system to minimize salt buildup. At least weekly, aspirate 50 to 100 ml of detergent solution (3 to 6 drops Triton X in reagent water) into the sample introduction system to minimize buildup of organic and other films on interior surfaces.

Lubricate the peristaltic pump rollers at least monthly.

### 3.2.2 Working Coil Cooling Water

Check the inlet air filter at the back of the recirculator at least weekly. Clean or replace as indicated by its appearance. Every six months drain and refill the recirculator according to the instructions in the operating manual for the unit. Add an organic algicide (e.g., Bardac 2250/2280) to the recirculator supply.

### 3.2.3 Data Files

At least weekly evaluate the space available on the data system hard drive using file management software. As indicated, transfer files to diskettes or tape. Maintain at least 10 megabytes of free space on the hard drive.

## APPENDIX H.6

### Preventative Maintenance, Gas Chromatograph/Mass Spectrometers

#### 1.0 Overall Maintenance

Overall maintenance on all GC/MS systems is covered by maintenance agreements with the manufacturer. That agreement provides for preventative maintenance visits and for all parts and labor necessary to keep the instruments operating according to specifications. Aside from the routine procedures described below, all service and maintenance of this equipment is performed by qualified technicians employed by the instrument manufacturer.

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

If properly maintained instrument fails to respond properly to startup routines or will not meet specifications when in operation, inform the supervisor and take steps to arrange for a service call.

#### 2.0 Maintenance Procedures

##### 2.1 Sample Introduction System

##### 2.1.1 Purge and Trap Autosampler (Tekmar 2000/2016)

Clean glassware is essential to proper operation of the autosampler. After every use glassware should be cleaned in accordance with the instructions given in the autosampler operating manual.

Check the system weekly (or more often as indicated by performance) for leaks. As indicated by the results of the test, replace glassware mounts, sample sparing needle fittings or other connecting fittings.

Replace the Tenax trap every six months (or more often as indicated by system performance), following the instructions given in the operating manual for the autosampler. Before use, condition new traps as specified in the operating manual for the autosampler.

If the system becomes contaminated by sample material, set the controller to bake for at least an hour. If prolonged baking does not eliminate the problem, it may be necessary to replace the Tenax trap. The procedure for replacing the trap is described in the operating manual for the sampler.

If the system becomes contaminated because an impure tank of purge gas was used, replace the hydrocarbon traps in the gas lines. If this is insufficient to restore the system to proper operation, the baking procedure described above may be required.

### 2.1.2 Direct Injection Autosampler (Hewlett Packard 7673)

Periodically clean the surface of the sampler tray arm, gripper, gripper jaws and tray quadrants. Do not use any lubricants on any part of these assemblies.

Periodically vacuum off any dust which has built up around the vents on the cover of the sampler controller.

Periodically remove the cover from the injector tower. Swing the needle guide arm out from behind the turret and clean the needle guide cone, needle support and nearby surfaces. Also clean the sample vial inserts on the turret and vacuum off any dust that builds up around the vents for the electronics assembly. Do not use any lubricants on any part of the injector tower.

As required, replace the vials for wash fluid and wastes in the tower assembly.

As required, replace the syringe and/or syringe needle in the unit following the instructions given in the operating manual.

## 2.2 Gas Chromatograph

### 2.2.1 Sample Inlet

At the end of each working day, replace the septum if the injections of standards or samples have been made.

Dependent on system performance, remove the injector linear following the instructions in the operating manual for the instrument. Clean the linear thoroughly according to the instructions in the operating manual for the instrument. After cleaning, deactivate glass liners using Sylon CT according to the instructions supplied with the material.

### 2.2.2 Columns

As indicated by system performance, remove the inlet and/or outlet ends of fused silica capillary or megabore columns and trim away several inches. The cut made should be inspected to ensure that it is smooth and at right angles to the column wall. Reinstall the column ends as specified in the instruction manual for the instrument and, after allowing an appropriate time for the system to equilibrate, recheck performance.

As indicated by system performance, it may be necessary to replace or repack the column. Remove the column from the instrument according to instructions in the operating manual. For packed columns, repack as specified by the instrument or column manufacturer.



Install the repacked column or the new fused silica column as specified in the instrument instruction manual. Condition the column as required before use. When conditioning is completed, reestablish the correct flows in the system and determine retention times as required.

## 2.3 Mass Detector

### 2.3.1 Vacuum System

All maintenance on the pumps, sensors and other components of this system is performed by qualified service technicians employed by the manufacturer.

### 2.3.2 Source

As indicated by system performance, vent the system and remove the source for cleaning. Disassemble, clean and reassemble the source following the procedures in the instrument operating manual. Reinstall the source and pump the system down. Verify that the system is ready for tuning, calibration and sample analysis as specified in the instrument operating manual.

## 2.4 Data System

The data system consists of three networked Hewlett-Packard RTE-A computers. All active data are stored on the disk drives of those computers.

Once a report has been submitted to the customer, a check of the files associated with that report is made. All of the raw data files associated with that report are downloaded to magnetic tape for long term storage. Finished data files (those associated with report forms) are not downloaded since a hardcopy of those documents is retained in the laboratory.

Taking one data system each week, a check of the files resident on the disk drive is performed. Those files which have been transferred to magnetic tape are marked for purging and a purge file is created and printed in hardcopy. The hardcopy is checked to verify that all files listed on it have been stored to tape. Subsequent to completing verification of the correctness of the purge file, the listed files are removed from the active system and the hard disk is packed.

The magnetic tapes are stored in a controlled environment. Storage time for each tape varies, dependent on ownership of the data it contains. Records are maintained indicating the date when storage time for a tape will expire. That date is determined based on data retention specifications of the customers involved.

## APPENDIX H.7

### Preventative Maintenance, Gas Chromatographs

#### 1.0 Overall Maintenance

Overall maintenance on all gas chromatographs is covered by maintenance agreements with the manufacturer. That agreement provides for preventative maintenance visits and for all parts and labor necessary to keep the instruments operating according to specifications. Aside from the routine procedures described below, all service and maintenance of this equipment is performed by qualified technicians employed by the instrument manufacturer.

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

If a properly maintained instrument fails to respond properly to startup routines or will not meet specifications when in operation, inform the supervisor and take steps to arrange for a service call.

#### 2.0 Maintenance Procedures

##### 2.1 Sample Introduction System

##### 2.1.1 Purge and Trap Autosampler (Tekmar 2000/2016)

Clean glassware is essential to proper operation of the autosampler. After every use glassware should be cleaned in accordance with the instructions given in the autosampler operating manual.

Check the system weekly (or more often as indicated by performance) for leaks. As indicated by the results of the test, replace glassware mounts, sample sparing needle fittings or other connecting fittings.

Replace the Tenax trap every six months (or more often as indicated by system performance), following the instructions given in the operating manual for the autosampler. Before use, condition new traps as specified in the operating manual for the autosampler.

If the system becomes contaminated by sample material, set the controller to bake for at least an hour. If prolonged baking does not eliminate the problem, it may be necessary to replace the Tenax trap. The procedure for replacing the trap is described in the operating manual for the sampler.

If the system becomes contaminated because an impure tank of purge gas was used, replace the hydrocarbon traps in the gas lines. If this is insufficient to restore the system to proper operation, the baking procedure described above may be required.

### 2.1.2 Direct Injection Autosampler (Hewlett Packard 7673)

Periodically clean the surface of the sampler tray arm, gripper, gripper jaws and tray quadrants. Do not use any lubricants on any part of these assemblies.

Periodically vacuum off any dust which has built up around the vents on the cover of the sampler controller.

Periodically remove the cover from the injector tower. Swing the needle guide arm out from behind the turret and clean the needle guide cone, needle support and nearby surfaces. Also clean the sample vial inserts on the turret and vacuum off any dust that builds up around the vents for the electronics assembly. Do not use any lubricants on any part of the injector tower.

As required, replace the vials for wash fluid and wastes in the tower assembly.

As required, replace the syringe and/or syringe needle in the unit following the instructions given in the operating manual.

## 2.2 Gas Chromatograph

### 2.2.1 Sample Inlet

At the end of each working day, replace the septum if the injections of standards or samples have been made.

Dependent on system performance, remove the injector liner following the instructions in the operating manual for the instrument. Clean the liner thoroughly according to the instructions in the operating manual for the instrument. After cleaning, deactivate glass liners using Sylon CT according to the instructions supplied with the material.

### 2.2.2 Columns

As indicated by system performance, remove the inlet and/or outlet ends of fused silica capillary or megabore columns and trim away several inches. The cut made should be inspected to ensure that it is smooth and at right angles to the column wall. Reinstall the column ends as specified in the instruction manual for the instrument and, after allowing an appropriate time for the system to equilibrate, recheck performance.

As indicated by system performance, it may be necessary to replace or repack the column. Remove the column from the instrument according to instructions in the operating manual. For packed columns, repack as specified by the instrument or column manufacturer.

Install the repacked column or the new fused silica column as specified in the instrument instruction manual. Condition the column as required before use. When conditioning is completed, reestablish the correct flows in the system and determine retention times as required.

## **2.3            Detectors**

### **2.3.1            Flame Ionization**

Periodically, or as indicated by system performance, inspect the jet and collector bore for cleanliness. Remove, disassemble and clean the unit following the instructions in the operating manual. While disassembled, inspect the jet and the various seals and gaskets for signs of wear or cracking. If damaged or worn, replace the defective parts.

Before attempting ignition of the flame, check the flow rates of hydrogen and air. If a major adjustment in the flow controller is required and the pressures in the supply lines are correct, disassemble the on-off valves as described in the operating manual for the instrument. Inspect the valves, valve stems and valve stem o-rings for cleanliness and/or signs of wear. Clean or replace as appropriate.

If proper flow rates have been established and the jet and base have been inspected and cleaned but the detector fails to ignite reliably, maintenance of the ignition system is required. Verify operation of the ignitor glow plug following the instructions in the operation manual for the instrument. As required, replace or adjust the glow plug, Teflon supporter or connecting wires or replace the fuse.

If further maintenance is required, consult a manufacturer's service technician.

### **2.3.2            Nitrogen-Phosphorus**

Periodically, or as indicated by system performance, inspect the collector assembly for cleanliness. Remove, disassemble and clean the unit following the instructions in the operating manual. While disassembled, inspect the internal parts and the various seals and gaskets for signs of wear or cracking. If damaged or worn, replace the defective parts.

Check the flow rates of hydrogen and air. If a major adjustment in the flow controller is required and the pressures in the supply lines are correct, disassemble the on-off valves as described in the operating manual for the instrument. Inspect the valves, valve stems and valve stem o-rings for cleanliness and/or signs of wear. Clean or replace as appropriate.

### **2.3.3            Electron Capture**

Correctly conditioned columns, carrier gas of ultra-high purity, cleanliness of injection ports and liners and freedom from leaks are all prerequisite to proper operation of instruments with these detectors. Rigid observance of these requirements as described in the instrument operating manual is fundamental to prevention of problems during operation. Procedures for checking gas purity and determining whether the system is leak-free are described

in the manual for the instrument. If problems occur, these procedures should be invoked as a first step in resolving the difficulty.

Periodically, or as indicated by system performance, bake out the detector following the procedures described in the system operating manual.

When none of the steps outlined above are effective in restoring a detector to proper operation it must be rebuilt by the manufacturer. Following the procedures outlined in the instrument manual, remove the detector assembly from the unit. Pack the assembly in a properly labeled container and ship it to the manufacturer for rebuilding.

Every six months perform a wipe test of the detector to check for radioactive leakage. Kits for conducting the test are available from the instrument manufacturer and the procedures for collecting the wipe samples are fully described in the instrument operating manual. Submit the wipe samples to an authorized agency for counting. Maintain continuing records of wipe test results.

#### 2.3.4 Electrolytic Conductivity

A cylinder (size 1A) of hydrogen reaction gas will last for approximately 1000 hours of operation. As indicated by pressure gauge readings on the cylinder regulator, renew the reaction gas supply.

The n-propanol used as conductivity electrolyte in the reservoir will slowly evaporate and the losses must be replaced. Check the level in the reservoir at least weekly. Top off the supply as required to maintain volume.

Every six months or after 2000 hours of operation, remove the roughing resin cartridge from the unit following instructions in the instrument operating manual. Open the cartridge and remove the resin. Following the procedures outlined in the instrument manual, liquid pack fresh resin into the cartridge. When packed, seal the cartridge closed and reinstall in the instrument.

#### 2.3.5 Photoionization

As required by system performance, replace the photoionization lamp following the instructions given in the instrument operating manual.

## APPENDIX H.8

### Preventative Maintenance, UV-Visible Spectrophotometers

#### 1.0 Overall Maintenance

Overall maintenance on all spectrophotometers is covered by maintenance agreements with the manufacturer. That agreement provides for preventative maintenance visits and for all parts and labor necessary to keep the instruments operating according to specifications. Aside from the routine procedures described below, all service and maintenance of this equipment is performed by qualified technicians employed by the instrument manufacturer.

Keep a record of all maintenance procedures in a dedicated logbook. Describe the maintenance activity briefly and sign and date the entry.

If a properly maintained instrument fails to respond properly to startup routines or will not meet specifications when in operation, follow the procedures noted below. If the problem is not resolved after taking these actions, inform the supervisor and take steps to arrange for a service call.

#### 2.0 Maintenance Procedures

At least monthly, check instrument operation using the standards provided for stray radiant energy, wavelength accuracy, 0% transmittance and photometric accuracy. If the instrument fails to respond appropriately to these standards or if difficulties are encountered in calibration or sample analysis, follow the trouble shooting procedures given in the operating manual. Take appropriate corrective actions.

If lamp replacement is required, align the new lamp using the procedures given in the operating manual.

After completing the required corrective actions, check instrument performance with the standards as described above.

After use, turn off all power to the instrument. Rinse interior and exterior surfaces of cuvettes with copious quantities of reagent water. Follow with a rinse of methanol or acetone. Invert the rinsed cuvettes and allow to drain dry. Store inverted in a covered container. Periodically wash all cuvettes thoroughly following the procedures outlined in Appendix A.4. Discard any cuvettes which are scratched, stained, cracked or chipped and replace with new.

Periodically wipe all exterior surfaces of the instrument with a damp cloth. Do not use abrasive cleaners or solvents since these may damage the finish.

## APPENDIX H.9

### Fume Hoods and Fire Extinguishers

Mean face velocity (sash fully open) of all hoods at ARDL is a minimum of 60 ft/min. (linear). This value is measured each month by a Dwyer vaneometer and recorded on Form QA-2. When unrestricted hood face velocity falls below the minimum, corrective action is taken immediately. To insure proper operation of exhaust hoods follow the routine noted below.

Monthly a check of the status of fire extinguishers in the laboratory are inspected. The reading on the indicator of the units is recorded on Form QA-12. Units which are not fully charged or are otherwise not to standard are brought to a state of readiness with appropriate corrective action.

# HOOD VELOCITY MEASUREMENT

MONTH: \_\_\_\_\_

Hood	Test	Test	Test	Test	Test	Test	Test
Test	Test	Test	Ft/Min				
Location	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	Point 7
Point 8	Point 9	Avg					

Front Lab

Hood 1

Main Water

Lab

Hood 2

Hood 3

Lab 1

Hood 4

Lab 2

Hood 5

Lab 3

Hood 6

Lab 4

Hood 7

Lab 5

Hood 8

Hood 9

Hood 10

Hood 11

Form

QA-2

Analyst



HOOD VELOCITY MEASUREMENT

MONTH: \_\_\_\_\_

Hood Location	Test Point 1	Test Point 2	Test Point 3	Test Point 4	Test Point 5	Test Point 6	Test Point 7	Test Point 8	Test Point 9	Ft/M Avg
------------------	-----------------	-----------------	-----------------	-----------------	-----------------	-----------------	-----------------	-----------------	-----------------	-------------

Hood 12

Hood 13

Hood 14

Hood 15

Hood 16

Hood 17

Hood 18

Hood 19

Hood 20

Form QA-2

\_\_\_\_\_  
Analyst

MONTHLY FIRE/SAFETY CHECK

MONTH: \_\_\_\_\_

Location	Fire Ext.	Eye Wash	First Aid Kit	Fire Blanket
Office #1, Bldg. #1				
H <sub>2</sub> O Lab Restroom				
Hallway/Computer				
Small H <sub>2</sub> O Lab				
Large H <sub>2</sub> O Lab				
Lab #1, Building #2				
Lab #2, Building #2				
Hallway/Building #2				
Lab #3, Building #2				
Lab #4, Building #2				
Hallway #2/Lab #5				
Lab #5/Door				
Lab #5/Wall				
Lab #6, Building #3				
Hallway/Lab #6/B #3				
GC Extraction Lab				
Ship/Rec'd Wall				
Ship/Rec'd Dbl Doors				
Hallway GC Room				
GC Room				
Hallway, Building #4				
Hallway of Building #4				
Office #1, Bldg. #4				
Building #4 Phone				
B. #4 Granulation Rm.				
Building #4, Backdoor				
Office #2, Bldg. #4				
Solvent Shed				
Production Room				

Comments:

Form QA-12

**ATTACHMENT 2**

**FIELD SAMPLING PLAN**

**ATTACHMENT 2**

**FIELD SAMPLING PLAN FOR**

**PHASE II SITE INVESTIGATION**

**CARUS CHEMICAL COMPANY**

**MANUFACTURING FACILITY**

**LASALLE, ILLINOIS**

Submitted to

State of Illinois  
Illinois Environmental Protection Agency  
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## 1. INTRODUCTION

### 1.1 Application and Scope

This Field Sampling Plan (FSP) describes the field procedures to be used in implementing the Phase II Site Investigation at the Carus Chemical Company manufacturing facility. All procedures discussed in this document will be conducted in compliance with the Work Plan, Quality Assurance Project Plan (QAPP), and the Site Health and Safety Plan (HASP). If deviations from the procedures outlined in this document are necessary, the alternative procedures, and the circumstances requiring their use, will be documented in the field book. The FSP is structured to address the following:

- *Organization*: Defines the field sampling organization.
- *General Field Procedures*: Describes the field procedures that will be used to collect data during the Phase II Site Investigation.
- *Sample Analytes, Handling Procedures, and Documentation*: Describes analytes, sample containers, volume, preservation, storage, handling, and documentation, shipment, chain of custody procedures, and data quality objectives for each sample type.
- *Decontamination Procedures*: Describes equipment and personnel decontamination procedures.

### 1.2 Organization

The field sampling organization will be comprised of engineers and geologists from GeoSyntec Consultants including Mr. Bryan Tindell and Mr. Jack Raymer, P.G. The sampling team will work with the drilling team members to extract samples while boreholes are being advanced.

### 1.3 General Field Documentation

General field procedures to be used in several of the tasks are addressed in this Plan. If deviations from these procedures are necessary, alternative procedures, and the reason for their use, will be thoroughly documented in the field book for that task. Due to the size and planned organization for this Phase II Site Investigation, it is anticipated that all site work documentation will be contained in one field book.

#### 1.3.1 Field Activity Logbook

The Field Activity logbook (referred to as the field book) will contain daily field activity information, site health and safety information, and equipment calibration information. The field book will be bound and have sequentially numbered pages. Entries will be signed and dated by the field personnel recording the information. Corrections to log entries will be made by crossing out incorrect entries with one line and initialing and dating the strike-out. The correct entry then will be made. At the end of each working day, the field book entry for that day will be copied and placed in the project file.

##### 1.3.1.1 Daily Field Activity Information

Task-specific activities will be recorded and will contain information regarding general site activities. Additionally, the Task Managers will use various forms for recording field data, such as chain-of-custody forms or monitoring well logs. Required daily field information will include:

- project number;
- on-site personnel;

- date and weather conditions;
- personnel leaving or arriving on site;
- time and description of task activities;
- description of any samples collected;
- record of drilling activities, including footage and supplies used; and
- description of well or piezometer installation, including calculations of filter pack, bentonite pellet seals, or grout volumes, and materials used.

Sampling events also will include the following information:

- date and time of sampling;
- sample medium and type (i.e., grab, composite, duplicate, etc.);
- sample collection technique(s);
- sample containers, analytes and preservatives;
- sample number, location, and depth;
- pertinent field observations;
- name of samplers;
- time of delivery of samples to lab or express courier; and
- photograph number and direction.

### 1.3.1.2 Site Health and Safety Information

Health and safety information for the various field activities will contain instrument calibration data and a record of the time, location, and concentration of contaminants measured during the various field activities. Readings from monitoring equipment will be recorded for each task on a daily basis, and will include the following information:

- signature of individual filling out information;
- date/time;
- names of personnel in exclusion zone and their affiliation;
- level of personal protection utilized;
- weather conditions;
- work activity/task;
- monitoring/sampling information: time, location of measurement (e.g., breathing zone, downhole), measurement value and units, and type of instrumental/serial number;
- calibration information: date and time of calibration, calibration results, type of instrument, and instrument serial number; and
- any other pertinent information with regard to health and safety (i.e., equipment entering exclusion zone and decontamination of equipment).

#### 1.3.1.3 Equipment Calibration Information

Equipment calibration data will be recorded for each piece of monitoring equipment used on site. The following information will be recorded:

- date and time of calibration;
- name of person conducting calibration;
- type of instrument and serial number;
- type of calibration conducted; and
- calibration results.

#### 1.4 Sample Collection Summary

A summary of all soil, sediment, ground-water, and surface-water samples to be collected during Phase II Site Investigation is shown on Tables 1 and 2 of the Work Plan. The preliminary locations of the piezometers are shown on Figure 2 of the Work Plan.

#### 1.5 Analyses

Samples will be analyzed selectively using the procedures outlined below. Procedures for constituents other than those proposed in Tables 1 and 2 are included in the event they are needed at a later date.

### 1.5.1 Volatile Compounds

Volatile compounds will be determined by EPA Method 8240. Method 8240 is a purge and trap method using the GC. The Toxicity Characteristic Leaching Procedure (TCLP) method will also be used to analyze soil and sediment samples. Duplicative samples shall be taken and aqueous samples will be preserved with HCl. Samples shall be kept at 4°C (39°F).

### 1.5.2 Base/Neutral and Acid Compounds

Base/neutral and acid compounds or semivolatile compounds will be analyzed using EPA Method 8270, except for polynuclear aromatic hydrocarbons (PAHs), which will be analyzed using EPA Method 8310. Method 8270 is a GC/MS method to determine 66 constituents. Samples will be iced from time of collection until extraction and extracted within 7 days for aqueous samples and within 14 days for soil samples. Samples shall be analyzed within 40 days following extraction. In addition, soil and sediment samples will be analyzed using the TCLP method. Method 8310 will be used to determine the concentration of PAHs.

Method 8310 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of 16 PAHs. Water and soil samples will be extracted within seven days and analyzed within 40 days. Samples will be iced from time of collection to extraction.

### 1.5.3 Pesticides and PCBs

Pesticides and polychlorinated biphenyls (PCBs) will be analyzed by EPA Method 8080. Method 8080 is a gas chromatograph (GC) method for the determination of 19 pesticides and 7 PCBs at the parts per billion level. Samples will be iced from time of collection until extraction. Water samples will be extracted within 7 days and analyzed within 40 days.

Soil samples will be extracted within 14 days and analyzed within 40 days. Additionally, soil and sediment samples will be analyzed using the TCLP method, EPA Method 1311.

#### 1.5.4 Inorganic Compounds

Inorganics will be analyzed using various techniques, including Inductively Coupled Plasma/Atomic Emission Spectroscopy, Atomic Absorption and Cold Vapor Atomic Absorption, depending on the inorganic compound and the matrix. All surface-water and ground-water matrices will be digested prior to analysis. In addition, solid samples (soils and sediments) will be extracted and analyzed using the TCLP method. The inorganics to be analyzed are listed in Table 2 of the Work Plan.

#### 1.6 Field Quality Control Samples

Field quality control (QC) samples will be collected in order to assess the potential for sample corruption during collection and handling and in order to evaluate the repeatability of the findings. Two types of QC samples will be collected and analyzed: (i) equipment blanks; and (ii) duplicates. This QC program is considered the most cost-effective means of preventing false positives resulting from field sampling procedures.

Equipment blanks consist of samples of the water used for decontamination after passing that water through decontaminated tubing, filter equipment, and other devices used in the sample collection process. If analytes are detected in equipment blanks, it indicates the possibility that those analytes may also have been introduced into other samples collected using similar procedures.

Duplicates are a complete second set of samples collected from selected sampling points. Duplicates are assigned separate and arbitrary

identification numbers from the original sample so that the laboratory has no indication that the sample is actually a duplicate. One duplicate sample will be collected for every ten samples collected. The determination of which samples will be collected in duplicate will be made by the Field Director. The duplicates will be chosen to represent a variety of the conditions encountered at the site. Equipment blanks will not be duplicated.



## 2. DATA QUALITY OBJECTIVES

### 2.1 General

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of data required to support decisions regarding remedial response activities. DQOs are determined based on the end uses of data collected; the data quality and level of analytical documentation necessary for a given set of samples will vary based on the intended use of the data. Five DQO Analytical Levels have been identified in the guidance document "Data Quality Objectives for Remedial Response Activities", OSWER Directive 9355.0-7B, March 1987. These DQO Analytical Levels and the types of data uses which may be appropriate for each level are outlined below:

- *DQO Analytical Level 1: Field Screening.* Appropriate for field screening or analysis using portable instruments. The results are often not compound-specific and quantitative, but results are available in real-time. Data indicates the presence or absence of measured parameters at sampling locations. This level is appropriate for site characterization monitoring (for example, an HNU photoionization detector used to monitor organic vapors during sample collection).
- *DQO Analytical Level 2: Field Analysis.* Characterized by use of portable analytical instruments which can be used on site or in mobile laboratories. A wide range of data quality can be generated from a mobile lab, depending upon the types of contaminants, analytical method and instrument used, calibration standards, sample preparation equipment, and training of the instrument operator. Both qualitative and quantitative data can be obtained in real-time or several hours. This level is appropriate for sample results used in site characterization, evaluation of remedial alternatives, engineering design, and monitoring during remedial action implementation. These mobile

instruments are generally limited to analysis of metals or volatile organic compounds.

- *DQO Analytical Level 3: Analysis in an Off Site Laboratory Using Methods Other than CLP Routine Analytical Services (RAS) Methods.* The analyses may or may not use CLP procedures, but do not use the validation or documentation procedures required of CLP Analytical Level 4 analysis. The laboratory may or may not be a CLP laboratory. Level 3 is appropriate for risk assessment, site characterization, evaluation of remedial alternatives, engineering design, and monitoring during implementation of remedial activities.
- *DQO Analytical Level 4: CLP RAS.* All analyses are performed in an off-site CLP analytical laboratory following CLP protocols. This level is characterized by rigorous QA/QC protocols and documentation and provides qualitative and quantitative analytical data. Level 4 is appropriate for risk assessment, evaluation of remedial alternatives, and engineering design activities.
- *DQO Analytical Level 5: Analysis by Non-Standard Methods:* All analyses are performed in an off-site analytical laboratory, which may or may not be in the CLP program. Method development or modification may be required for specific constituents or detection limits. The amount of documentation available for Level 5 analytical support will vary depending on the sophistication of the technology used. Level 5 is appropriate for risk assessment and possibly site characterization and evaluation of remedial alternatives, when contaminants at the site cannot be analyzed by conventional methods.

## 2.2 Proposed DQO Levels

Soil, sediment, surface-water and ground-water samples collected during the Phase II Site Investigation will be analyzed and validated using Data Quality Objective (DQO) Analytical Level 4.

### **3. MONITORING WELL SAMPLING**

#### **3.1 New Monitoring Wells**

New monitoring wells will be installed and sampled during the Phase II Site Investigation. These new wells will be constructed and installed in accordance with Section 4.8 of the Work Plan.

##### **3.1.1 Determination of Casing Volume**

Casing volumes will be determined by measuring the water level in the well and utilizing well construction data to calculate the well's casing volume. A decontaminated water-level indicator will be used to record the depth from the open top of the well casing (riser) to the top of the water surface to the nearest 0.10 of a ft (0.03 m). The water level indicator will be decontaminated with an alkanox wash and distilled/deionized water prior to each measurement. This information, along with well construction (depth of well) data from the new and existing wells, will then be used to calculate the volume of standing water in each well. This volume is referred to as a casing volume. The measurement will be taken with a decontaminated weighted tape and recorded to the nearest 0.10 ft (0.03 m).

##### **3.1.2 Well Development/Purging**

Well development will be performed in accordance with Section 6.4 of this FSP. Purging will be accomplished by using either laboratory cleaned bailers, a peristaltic pump, or a centrifugal pump. All tubing used in conjunction with the pumps will be decontaminated. Once three casing volumes have been removed, pH, specific conductance and temperature measurements will be taken in accordance with Section 3.3 procedures. Field instrument readings for these three parameters will be taken repeatedly during continued purging. When readings for these

parameters have stabilized and a minimum of three casing volumes have been purged, or a maximum of five casing volumes purged, a sample will be collected for laboratory analyses. Readings will be considered stable when successive readings are within 5% of each other. The field instruments and field sampling equipment will be washed in an alkanox solution and rinsed with distilled/deionized water between measurements. Instrument readings will be recorded in the field notes.

### 3.1.3 Well Sampling

Ground-water sampling will be performed within two hours of well development or purging (if sampling is not performed following well developemnt), after the well has stabilized and recharged. Sampling will be performed with laboratory clean, Teflon®-lined, single check valve (bottom) bailer, or by a peristaltic pump. Each bailer will have a dedicated Teflon®-coated stainless steel leader that will be decontaminated along with the bailer. The leader will be equivalent in length to the bailer but not longer than twice the bailer length. The leader will be connected to a nylon cord for sampling or purging. The nylon cord will be replaced with new nylon cord prior to initiating a sampling or purging event on another well.

In the event that recovery time of a well is greater than 24 hours, samples will not be taken. If the well is incapable of producing a sufficient volume of water to complete the minimum volume requirements of all sample bottles, any sample bottles already filled will be emptied into the drums or tanks holding the purged water and will be treated along with purge or well development water. VOC samples will always be collected first, directly from the first bailer and the septum vial will be free of bubbles or headspace.

### 3.2 Field Measurements

#### 3.2.1 Overview

Temperature, pH and specific conductance field measurements will be performed on grab samples taken during purging and on grab samples taken during ground-water sampling. Grab sample measurements will be placed in a decontaminated beaker. All field instruments and sampling equipment will be decontaminated with repeated alkanox solution washes and distilled/deionized water washes and rinses between well measurements. The following paragraphs describe the calibration procedures for the field measurement instruments.

#### 3.2.2 pH

The pH meter must be calibrated a minimum of twice each day using at least two different laboratory supplied pH solutions. This calibration consists of the following steps:

- Immerse the probe in the buffer solution. Perform the initial calibration.
- Rinse the probe, with distilled/deionized water and perform the second calibration on the neutralized pH solutions.
- Record in the field book what buffer solutions were used.
- When the meter is moved to a new location, recheck pH calibration by measuring the pH value of the buffer solution closest to the expected range of the sample.
- If unacceptable deviations occur, consult the operating manual for the appropriate remedial course of action.

### 3.2.3 Specific Conductance

The specific conductance/thermistor meter is not likely to exhibit random fluctuations. Daily calibration checks with a standard KCl solution, within the expected conductivity range of the water being measured, will be performed by:

- immersing the probe into the standard solution and performing initial calibration of the instrument; and
- thoroughly rinsing the probe with distilled/deionized water after immersing in KCl standard solution.

In addition to daily checks for the conductivity readings, the thermistor readings also must be checked daily. This is accomplished by taking a temperature reading of the KCl standard solution while the conductivity probe is immersed. Since specific conductance is temperature-dependent, instrument readings will be adjusted accordingly.

### 3.3 Sample Identification

Sample identification will be accomplished as follows:

- Each sample bottle will be labelled, tagged and numbered.
- Lids will be screwed on securely.
- Collector's initials and date will be marked on each label.
- All required information will be recorded in the field book, Chain-of-Custody form, sample tag, laboratory label, and on the sample log sheet.

Each sample will be uniquely identified by a six-digit number. The first number will represent the year (e.g., 3 for 1993), the next three numbers will represent the particular day of the year (e.g., 236 for 24 August) and the last two numbers will represent the sequential number of the sample taken on that day.

### 3.4 Sample Location Marking

Each new well will be identified as noted in Section 6.5. The existing wells will be identified by their earlier designation. Each well will be readily identifiable as the source of the sample.

### 3.5 Decontamination Procedures

Field instruments, bailers and other field sampling equipment will be decontaminated by rinsing with distilled/deionized water, washing with an alkanox wash and rinsing with a final rinse in distilled/deionized water. Sampling equipment will be wrapped in aluminum foil for transport to the next sampling point.

### 3.6 Sample Custody

Samples will be packed for shipping as directed in Section 7. A custody seal will be attached to each shipping package, and Chain-of-Custody forms will be properly filled out and enclosed or attached.

Samples will be returned to the drop area designated by the Task Manager and custody will be transferred using the Chain-of-Custody form to the Task Manager or other person designated to perform additional sample packing and shipping as detailed in Section 7.



### 3.7 Sample Filtering

When analyzing for metals, ground-water samples will be filtered if the sample is characterized by a turbidity of 5 nephelometric turbidity units (NTUs) or greater. The ground-water sample will be filtered in accordance with the draft USEPA recommendation dated 30 June 1992 using a 5  $\mu\text{m}$  cellulose nitrate filter, if readily available from suppliers. The in-line filter will be placed between the collected water and the sample container. If a 5  $\mu\text{m}$  filter is not available, a 0.45  $\mu\text{m}$  cellulose nitrate filter will be used and filtered and nonfiltered samples will be obtained.

#### **4. SURFACE-WATER AND SEDIMENT SAMPLING**

##### **4.1 Surface-Water and Sediment Sampling**

###### **4.1.1 Surface-Water Sampling**

Surface-water samples will be taken in accordance with the Work Plan. Each sample will consist of a sufficient volume to fill the required sample containers. All sampling equipment will be decontaminated after each sampling event as per Section 4.1.5.

###### **4.1.2 Field Measurements**

Field measurements will be taken for pH, specific conductance, and temperature and recorded in the bound field notebook at each of the sampling locations. Field instrument calibration and operations will be in accordance with Section 3.3. Pertinent observations, such as color or odor, also will be noted. Field measurement equipment will be decontaminated between sampling points using distilled/deionized water.

###### **4.1.3 Sample Identification**

After the cap is screwed tightly on the sample container, the completed label and sample tag will be attached to the container. The sample label will be marked on each container using a permanent marker. All required information will be recorded in the field book, Chain-of-Custody form, label, and on the sample log sheet.

###### **4.1.4 Sample Location Marking**

Surface-water and other sampling locations will be marked for future reference and subsequently tied into the survey grid system. A wooden

stake will be driven into the ground at the sampling point and a marker will be attached identifying the sample number and sampling point. If the marker is driven into the bank near the sampling location, the distance and direction of the actual sampling point from the stake will be noted in the field book. The distance from the stake to two survey points then will be measured. Distances will be recorded on a reference grid and the appropriate grid will be identified in the field book.

#### 4.1.5 Decontamination Procedures

As noted in previous paragraphs, field measurement devices will be decontaminated with an alkanox wash and rinsed with distilled/deionized water between sampling points. Pond samplers, miscellaneous sampling tools, trowels, and bucket augers will be decontaminated by:

- tap water wash with non-phosphate soap;
- tap water rinse and distilled/deionized water rinse;
- 10 percent nitric acid rinse (for samples to be analyzed for metals only);
- distilled/deionized water rinse;
- alkanox rinse;
- air dry, (totally); and
- distilled/deionized water rinse.

Decontaminated sampling equipment will be wrapped in aluminum foil for transfer to the next sampling point.

#### 4.1.6 Sample Custody

Samples will be returned to the drop area designated by the Field Engineer and/or Geologist and custody will be transferred using the Chain-of-Custody to the Laboratory.

## 5. SOIL SAMPLING

### 5.1 Sample Number and Locations

Subsurface soil samples and sediment samples will be collected to aid in characterizing the site. The subsurface will be characterized through analysis of soil and slag samples collected from a split-spoon sampler in conjunction with the construction of new monitoring wells and advancement of soil borings. Thin-walled (Shelby) tube sampling may also be performed to characterize the site.

Split-spoon samples will be taken from borings associated with new and replacement monitoring wells. The location of the borings and their respective well identification numbers are presented in the Work Plan. At locations where multiple wells may be installed, only the deeper boring will be sampled. Table 1 of the Work Plan presents a summary of the borings and estimated depth of each sample and boring. The matrix and number of samples to be extracted from each boring is also noted. The split-spoon samples will be recovered at 5-ft (1.5-m) intervals throughout the borings and at observed changes in stratigraphy.

### 5.2 Sampling Procedures

Standard penetration tests will be conducted as per standard methods, ASTM D1586-67 (1974). This procedure employs a split-spoon sampler and will be used to obtain representative samples of soil for visual classification and laboratory testing, and to obtain a measure of the resistance of the soil to penetration by the split-spoon sampler.

Subsurface material will be obtained at 5-ft (1.5-m) intervals using a split-spoon sampler. The material will be removed from the split-spoon sampler with a decontaminated stainless-steel spatula and placed in a laboratory bottle. Care will be taken to ensure that the sample is not mixed prior to placing the material in the sample bottle in an effort to

prevent loss of volatiles into the atmosphere. Each bottle will be capped tightly and labeled on both cap and jar with the sample number, borehole number, depth, number of jars per split-spoon sample, date, and sampler's initials.

The split-spoon sampler will be decontaminated after sampling. A minimum of three split spoons will be available during sampling to minimize delays related to the decontamination procedures and expedite the sampling. Liquid waste from the decontamination of the split-spoon sampler will be collected and disposed of with other well development water as per Section 6.7.1.

A clean, flat, smooth work surface (4 ft by 8 ft (1.3 m by 2.6 m) minimum), covered with 6-mil polyethylene sheeting, will be provided at each boring location. All disassembly, washing and assembly of the split-spoon samplers and Shelby tubes will be done on the above mentioned work surface.

### 5.3 Field Measurements (Head Space Screening)

The concentration of volatile organic compounds that collects in the head space of the designated standard glass sample jar from each split-spoon sample set will be measured in areas of concern. The head space analyses will be completed at the boring location site. The designated head space sample jar will be allowed to stand for at least 10 to 15 minutes to adjust to ambient temperature. After adjustment occurs, the designated sample jar will be uncapped, the aluminum foil seal punctured with the instrument's probe, and a sample extracted from the sample jar's head space. The sample will be extracted by using the probe and pump system within the appropriate instrument. Instrument readings will be recorded in the field book.

Field records of the screening results will be kept for each boring to indicate borehole number, depth and instrument readings.

#### 5.4 Sample Identification

Sample jar lids will be hand tightened. The label and sample tag will be completed and attached to the jar. The sampler will initial and date the label and mark the sample depth on each of the sample jars using a permanent marker. The jars will be placed in a labeled sample jar box.

All required information will be recorded in the field book, and on the Chain-of-Custody form, sample tag, label and on the sample log sheet.

Laboratory samples and remaining material sampled from the test boring will be returned to the drop area designated by the Task Manager and custody will be transferred using the Chain-of-Custody form. Sample packing and shipping details are provided in Section 7.

Health and safety precautions during test borings and sampling will be in accordance with the Site HASP.

Soil samples will be identified by monitoring well/boring number and measured depth from the ground surface. Drill logs will be maintained throughout drilling and sampling procedures.

#### 5.5 Decontamination Procedures

All augers and other drilling tools in contact with the soil samples will be thoroughly steam cleaned prior to use at each boring. At the completion of the test boring field investigation, the drill rigs, other equipment and all tools will be steam cleaned prior to leaving the site. Field instruments, sampling tools and measuring devices will be decontaminated as described in Section 4.1.5.

## 6. MONITORING WELL AND PIEZOMETER CONSTRUCTION, INSTALLATION, AND DEVELOPMENT

### 6.1 Construction

Monitoring wells will be constructed under the direction of a professional engineer or geologist. New monitoring wells will be constructed by utilizing the hollow-stem continuous flight auger method with split-spoon samples taken typically on 5-ft (1.5-m) intervals. A summary of the proposed piezometers are shown in Table 1 of the Work Plan. The Boring Log and Monitoring Well Log forms are provided as Appendix A to the FSP.

Borings to be used for the installation of the wells will be constructed utilizing the hollow-stem continuous-flight auger method. In all of the piezometers and shallow wells, the borings will be advanced to a depth that will allow the top of the well screen to be installed at or below the seasonal high water table elevation, provided a sufficient amount of water exists above the bedrock.

Borings advanced utilizing the hollow-stem continuous-flight auger method will leave the augers in place during split-spoon sampling and during the initial steps of monitoring well installation. The augers are left in-place to provide lateral support to maintain the borehole opening, while split-spoon sampling and well installation is performed through the hollow-stem portion of the augers. As well installation progresses, the augers are gradually removed and the well installation backfill materials are placed within the well annulus.



## 6.2 Monitoring Well Construction Materials

Well construction materials will be durable enough to resist chemical and physical degradation and will not interfere with the quality of ground-water samples. Section 4.8.3 of the Work Plan describes the various materials to be used in the construction of the Phase II Site Investigation monitoring wells.

## 6.3 Installation and Abandonment Procedures

### 6.3.1 Overview

Monitoring wells will be installed under the direction of a professional geologist and will be installed in accordance with IEPA policies and procedures. Installation of the wells will occur after the advancement of boreholes. Borings will be advanced by utilizing either the continuous-flight hollow stem auger method or the mud rotary method as described in the previous sections. Borings will be advanced to depths indicated in Table 1 of the Work Plan.

Borehole cuttings will be drummed and miscellaneous debris removed from the immediate area prior to the initiation of well installation activities. Volume estimates will be calculated for individual well construction materials and adequate quantities of those materials prepared (decontaminated) and brought to the well construction site.

### 6.3.2 Well Screen and Riser Installation

Well screen and riser pipe for the shallow monitoring wells will be installed according to the following sequence:

- 6 to 8 in. (152 to 203 mm) of filter pack material will be placed through the hollow stem portion of the augers;

- a PVC end cap will be placed onto the lowermost section of well screen;
- a manageable section of well screen and riser assembly will be inserted through the hollow stem portion of the augers; and
- additional sections of riser assembly will be added so that the completed assembly rests on the filter pack material and extends above the ground surface.

All pieces of the riser assembly will be steam cleaned prior to installation and care will be taken during handling and installation to ensure that contamination does not occur. Riser pipe material will not be laid on the ground. Work stands with decontaminated surfaces will be used during handling, storage and installation. Tools and rigging in direct contact with riser assembly parts will be decontaminated by steam cleaning prior to initiating installation procedures at each well. Crew members and staff in contact with the riser assembly parts will use clean gloves when handling the riser assembly.

### 6.3.3 Filter Pack Installation

Filter pack material will be added to the well annulus area between the hollow stem auger or the mud rotary borehole and the riser pipe assembly. As noted before, the appropriate quantity of material will be calculated and measured prior to initiating installation. Elevation measurements will be taken by using a decontaminated weighted tape measure to ensure that the filter pack extends to above the top of the well screen, but not in excess of two feet above the top of the well screen. The quantity estimates and measurements will ensure that bridging does not occur or will be detected within the filter pack and that the filter pack covers the well screen. In the shallow wells, the augers will be pulled slowly as materials are added to the annular area to ensure that the annular area remains open during installation

procedures. In the deeper wells, the filter pack will be placed in the annular space between the well casing the borehole wall.

#### **6.3.4 Bentonite Seal Construction**

Certified sodium bentonite pellets will be added to the annular area above the filter pack. The pellets will be tamped into place when possible. If the bentonite seal is above the water table, 5 gal (19 l) of tap water will be added to aid in the swelling and sealing effect of the pellets. The bentonite seal will be pre-measured and post-measured to ensure that a continuous three foot thick layer of bentonite is installed. As with the filter pack installation, the augers will be pulled slowly as the bentonite pellets are added to allow the pellets to fill the well annulus between the borehole's outer perimeter and the riser assembly.

#### **6.3.5 Well Construction Above the Bentonite Seal**

High-solids bentonite grout or bentonite in the dry state will be placed in the annular space above the bentonite seal. If dry bentonite is required due to the porosity of the slag, sufficient potable water will be added to ensure swelling of the bentonite. The grout or dry bentonite will be placed from the top of the bentonite seal upward to the estimated frost line. The augers will be slowly pulled as the grout or dry bentonite is added. All equipment will be steam-cleaned prior to use at the site.

#### **6.3.6 Sealing of Borings**

Borings will be sealed in accordance with IEPA protocol. The materials used to seal the annular space will be chosen and installed in an attempt to prevent migration of contaminants (if existing) from one

strata to another. The materials will be chemically compatible with the anticipated waste (if present) to ensure seal integrity. Within a given strata, chemically inert or natural materials of the same or lesser hydraulic conductivity may be placed within the borehole provided that there is no danger of cross-contamination between strata.

#### 6.4 Development

All new monitoring wells will be developed by pumping and flushing formation waters through the completed well until the natural hydraulic conductivity of the formation has been restored and all foreign sediment removed. The flow of development waters may be reversed frequently to reduce the possibility of bridging. All development equipment will be steam cleaned prior to the initiation of a well development activity. Wells will be developed until the discharge is turbid free and successive specific conductance field readings have stabilized over a five minute period.

#### 6.5 Security and Identification

Risers will be capped with a PVC threaded vented cap that can be removed for sampling. A 5-ft (1.5-m) length of 6-in. (152-mm) diameter protective casing will be set and cemented in-place as part of the well security system. A locking cap with a padlock will complete the well security system. All padlocks will be a single key master set. The protective steel casing will be wrapped with highly visible plastic tape. Preferred colors are highly visible oranges, yellows or reds.

#### 6.6 Documentation

The following information will be compiled and documented in the field book:

- date/time of construction;
- drilling method;
- approximate well location and surface elevation;
- borehole diameter and well casing diameter;
- well depth ( $\pm 0.1$  ft (30 mm)) and casing length protruding above ground surface;
- drilling and lithologic logs;
- casing material;
- screen materials and design;
- casing and screen joint type;
- screen slot size/length;
- filter pack material;
- filter pack volume calculations;
- filter pack placement method;
- sealant materials;
- sealant volume;
- sealant placement method;
- surface seal design/construction;

- well development procedure;
- type of protective well cap;
- estimated ground surface elevation;
- estimated top of casing elevation; and
- detailed drawing of well (include dimensions).

## 6.7 Hydrogeologic Testing

### 6.7.1 Slug Test Procedures

A cylindrical object of known volume (a slug) is lowered into or removed from a well instantaneously. The water level in the well is monitored periodically from that instant until the time the water level returns to its original level before instantaneous insertion or removal. Measurements of the water level in the well will be taken immediately prior to instantaneous insertion or removal of the slug, immediately after the instantaneous insertion or removal of the slug and at regular intervals thereafter, until the water level returns to its original measurement for three consecutive readings. Water level readings and time of the read will be recorded with an In-Situ® SE1000B Data Logger or equivalent. Shallow wells will be tested under rising of well screen material head conditions (instantaneous removal of the slug) due to the location of well screen material, 2 ft (0.6 m) above the encountered water table.

### 6.7.2 Decontamination

All materials in direct contact with the formation water will be decontaminated prior to each slug test in accordance with Section 4.1.5

criteria. Nylon cord used to retrieve the slug will be replaced prior to each slug test.

## 7. SAMPLE PACKING, SHIPPING AND DOCUMENTATION

### 7.1 Sample Packing

Each sample container will be placed in an individual plastic bag to prevent cross-contamination during shipping. All aliquots of the surface-water, ground-water, soil, or sediment samples will be placed in shipping coolers with enough ice to maintain a temperature of 4°C and sufficient vermiculite or bubble pack to prevent breakage during shipping. All samples believed to be heavily contaminated will be shipped in coolers separately from other samples. Water and soil samples to be analyzed for metals will be shipped to the laboratory in the same coolers as the organic samples even though these samples do not require ice for preservation. All sample aliquots in a shipping container will be listed on the chain-of-custody enclosed in the container. Once the samples are securely packaged, the container will be sealed with strapping tape and several custody seals will be placed over the top edge.

### 7.2 Sample Shipping

Samples will either be picked up from the site by a ARDL Laboratory representative, delivered to ARDL by a member of the site project team or shipped via overnight service to ARDL.

### 7.3 Sample Documentation

Documents recording sampling events will include daily entries into the field book, sample labels, and chain-of-custody seals and forms. The information to be recorded for each sampling event is discussed in Section 1.2.



### 7.3.1 Sample Labels

Each sample bottle will be labelled with the following information: date and time of sample collection, sample number, analyte(s), preservative, project and task number, and sampler's initials. Indelible ink will be used to record information on the sample label.

### 7.3.2 Custody Seals

Custody seals will be used when a sample shipment is picked up by the laboratory or sent to the laboratory by a third party carrier. Signed and dated custody seals will be attached to the top of the shipping container in such a way that it is necessary to break the seal to open the container. Custody seals ensure that any tampering during transportation will be detected by the receiving laboratory.

### 7.3.3 Chain-of-Custody Forms

Chain-of-custody forms provide the documentation to trace sample possession from the time of sample collection until receipt by the laboratory. One chain-of-custody form will be filled out for each cooler or shipping container and will list all the sample aliquots contained in the cooler or container. An example chain-of-custody form is presented in ARDL (November 1992). One copy of the completed form will be placed in a plastic bag taped to the inside lid of the shipping container, one copy will be kept with the project files, and one copy will be sent to the Field Engineer.

## 8. SEDIMENT MAPPING

### 8.1 Introduction

Sediments from the holding pond will be mapped in three dimensions by collecting core samples at various sampling points using a piston sampler and probe rods. Samples will be collected from a boat. Sampling locations will be controlled by a sampling grid. In cases where the samples will be analyzed for constituents of concern, equipment will be decontaminated in accordance with the procedures presented in Section 4.1.5 of this document. The remainder of this section consists of descriptions of the three primary elements of sediment mapping: (i) layout of the mapping grid; (ii) sediment probing; and (iii) sediment coring.

### 8.2 Mapping Grid

A mapping grid will be established across the holding pond for the purpose of accurately locating sampling points. The mapping grid will be established by traversing the area above the pond with ropes, from east to west. The ropes will be secured to trees, other available appurtenances, or driven stakes. After the sediment mapping task is completed, the grid will be removed.

The ropes will be used as described below:

- The ropes traversing the holding pond will be assigned an identification number. The identification numbers and relative location of the grid elements will be recorded in the field book.
- The trees, other available appurtenances, or driven stakes which are used to establish the grid will be marked with flagging tape. The grid identification will be written on the tape with indelible ink.

- The magnetic-north bearing of each rope will be measured and recorded in the field book.
- The distance to each sample location will be measured along the rope from the securing structure. The distance will be recorded in the field book.

### 8.3 Thickness Probing

Steel rods will be driven in the sediment at selected locations in order to identify the thickness of the sediment. The rods will be constructed so that additional lengths of rods can be screwed onto the end above the holding pond to achieve sufficient length to reach the stratum beneath the sediment. It is assumed that the stratum beneath the sediment will be hard slag. After use, the steel rods will be removed. The following information will be recorded for each probe:

- the location of the probe in terms of rope identification and distance from the securing structure;
- the depth below water to the top of the sediment; and
- the depth below water to the top of the substratum.

### 8.4 Piston Coring

Piston cores will be collected from selected locations. The piston cores will be used for the following purposes: (i) visual description; (ii) sample collection for chemical analysis; and (iii) sample collection for permeability analysis.

The core barrel will consist of nominal 2-inch diameter, rigid plastic pipe with flush joint threads. The flush-joint threads will

enable pipe to be added to the core barrel. If possible, the plastic will be clear. A piston will be constructed to fit snugly within the core barrel. A rope will be attached to the back side of the piston.

The core barrel will be advanced by pushing it or driving it into the sediment. As the barrel is advanced, the piston will be gently pulled back with the rope to cause a slight suction to develop within the core barrel. Samples will be removed from the core barrel by pushing the piston with a rod.

In cases where the samples are to be analyzed for permeability, the section of the core barrel containing the interval to be analyzed will be cut out from the barrel with a saw. The cut section will have a minimum length of 1 ft (300 mm). The ends of the cut section will be sealed with wax in similar manner as general Shelby tube procedures. The up direction will be clearly marked on the core barrel so that the sample can be extruded in the same direction as it was collected.

## 9. RIVER-FLOW MEASUREMENTS

Flow in the Little Vermilion River will be determined at the points designated in the Work Plan using the stream profiling method. The stream profiling method consists of two parts: (i) measurement of the velocity of the stream; and (ii) measurement of the cross-sectional area of the stream. The total flow at the measurement point is obtained by integrating velocity over cross-sectional area.

Stream velocity will be measured using a current meter. Measurements will be made at varying depths and at several locations across the traverse of the stream. The stream profile will be made by measuring the depth of the water at various points across the width of the stream using a ruler. A detailed drawing of the cross section of the stream will be made on the basis of measurements and observations. The locations of the velocity measurements will be indicated on the cross section.

**APPENDIX A**

**BORING LOG**

**AND MONITORING WELL LOG FORMS**

**GEO SYNTEC CONSULTANTS**

Boca Raton, Florida

BORING  
START DATE  
FINISH DATESHEET OF  
ELEVATION  
DATUM

PROJECT

LOCATION CARUS CHEMICAL COMPANY / LASALLE, IL  
NUMBERGS FORM:  
AD1 12/91 10/93**BORING LOG**

DEPTH (FT)	MATERIAL DESCRIPTION	DEPTH (FT)	SAMPLES				COMMENTS
			NUMBER	REC (%) OR REC & RQD (%)	SPT VALUES		
					BLOWS PER 6"	N	
0.0		0					
5.0		5					
10.0		10					
15.0		15					
20.0		20					
25.0		25					
30.0		30					
35.0		35					
40.0							

TRACTOR LAYNE ENVIRONMENTAL  
MILLER  
DRILL RIG  
DIA/METHOD  
LOGGER

NORTHING  
EASTING  
ANGLE Vertical  
BEARING -----  
PRINTED 20 OCT 93

REMARKS:

SEE KEY SHEET FOR SYMBOLS AND ABBREVIATIONS.

**GEO SYNTEC CONSULTANTS**

Boca Raton, Florida

BORING  
START DATE  
FINISH DATE

WELL

ELEVATION  
DATUM

SHEET OF

PROJECT

LOCATION CARUS CHEMICAL COMPANY / LASALLE, IL  
NUMBERGS FORM:  
WELL1 3/91 8/90**MONITORING WELL LOG**

LAYER DEPTH (FT)	MATERIAL DESCRIPTION	SYMBOLIC LOG	WELL LAYER DEPTH (FT)	WELL COMPONENTS	COMMENTS
				ELEVATIONS (FT): RISER: COVER:	
5.0					
10.0					
15.0					
20.0					
25.0					
30.0					
35.0					
40.0			40.0		

CONTRACTOR LAYNE ENVIRONMENTAL

RILLER

DRILL RIG

DIA/METHOD

LOGGER

NORTHING

EASTING

ANGLE Vertical

BEARING

PRINTED 20 OCT 93

REMARKS:

SEE KEY SHEET FOR SYMBOLS AND ABBREVIATIONS.



APPENDIX C



## **APPENDIX C**

### **RÉSUMÉS**

**NEIL D. WILLIAMS**

**hazardous waste management,  
solid waste management,  
geosynthetics,  
geotechnical engineering,  
contaminant transport**

## **EDUCATION**

University of California, Berkeley: Ph.D., Geotechnical Engineering, 1982

Utah State University: M.S., Geotechnical Engineering, 1979

Utah State University: B.S., Civil/Structural Engineering, 1977

## **PROFESSIONAL HISTORY**

GeoSyntec Consultants, Boca Raton, Florida, Chief Executive Officer, President, Principal

Georgia Institute of Technology, Atlanta, Georgia, Assistant Professor, 1984 - 1988

Law Environmental, Inc., Atlanta, Georgia, Senior Engineer, 1984 - 1988

Woodward-Clyde Consultants, Overland Park, Kansas, Project Engineer, 1982 - 1984

University of California, Berkeley, California, Research Assistant, 1980 - 1982

Utah State University, Logan, Utah, Instructor, 1978 - 1979

Civil Engineering Research Facility/UNM, Albuquerque, New Mexico, Research Engineer,  
1977 - 1978

## **REPRESENTATIVE EXPERIENCE**

Dr. Williams has performed research and consulting in the Hazardous and Solid Waste Management areas for over 10 years. These activities include design, remediation, or closure of over 40 landfills and surface impoundments, preparation of permits for RCRA facilities, development of contingency plans and ACL petitions, remedial investigations, hydrogeologic assessments, feasibility studies and design of ground-water recovery and treatment systems. Dr. Williams has also developed numerical models to evaluate contaminant transport, recovery system efficiency, and the transport and recovery of pure phase liquid in both the saturated and unsaturated zones. In addition, Dr. Williams has provided expert testimony for several industrial clients on waste management facilities.

Dr. Williams has also performed research and consulting relating to soil/chemical interactions. These analyses led to the design of clay liners and slurry walls for hazardous waste management facilities and clean-up projects. Dr. Williams has developed equipment and analytical methods

to evaluate changes in soil properties such as conductivity, density, void ratio and Atterberg limits as well as transport and absorption characteristics of chemicals in various soils.

Dr. Williams has developed considerable expertise in the design and implementation of recovery systems for nonaqueous phase liquids (NAPLs). He has developed two numerical models to evaluate the transport of NAPLs in soil. These models have been used to evaluate the cost effectiveness of remedial alternatives for NAPL recovery systems. Based on these analyses, Dr. Williams has designed innovative and cost effective recovery systems for 3 refineries and over 20 facilities. Due to his experience in remedial investigations and hydrogeologic assessments as well as design and evaluation of treatment and recovery systems, Dr. Williams has a unique ability to focus available resources and find innovative and cost effective solutions to complex problems.

While at the Georgia Institute of Technology, Dr. Williams developed new course materials for "Designing with Geosynthetics", "Geotechnical Considerations in Hazardous Waste Containment", "Soil Plasticity", "Numerical Methods in Geotechnical Engineering", and "Advanced Soil Mechanics". Dr. Williams also performed research on in-situ bioremediation, leachate generation in municipal solid waste and codisposal landfills, started the Geosynthetics Research Laboratory, and initiated over 20 research projects related to the use of geosynthetics. The primary geosynthetic-related research activities included design and evaluation of double liners for RCRA facilities, development of analytical methods for evaluation of slope stability in RCRA facilities, evaluation of the filtration characteristics of geotextiles in soil, evaluation of soil/geosynthetic interface friction, evaluation of soil/geosynthetic interaction pullout, development of new constitutive equations for interface elements, slope stability of reinforced slopes and walls, design of embankments with base reinforcement, evaluation of synthetic drainage composite flow properties, and evaluation of tension and compression creep of geosynthetics.

While a Research Assistant at the University of California, Dr. Williams performed research on the containment of high level radioactive wastes in seafloor sediments. This included the design and assembly of a high temperature triaxial compression device, performing laboratory tests on seafloor sediments at elevated temperatures (4 to 200 degrees centigrade) and pressure, and the development of constitutive equations and analytical models required to describe sample deformations due to specific thermal and stress histories.

Prior to obtaining his Ph.D. degree, Dr. Williams was a Research Engineer for two years with the Civil Engineering Research Facility in Albuquerque, New Mexico. During this time, he performed research work related to the design of laboratory equipment to test the deformation characteristics of partially saturated soils subject to the positive and negative air pressure pulses resulting from the surface detonation of high explosives; the design and assembly of a remote sensing aerial reconnaissance system for bomb damage assessment; the design of Radome

Coverings to withstand projectile perforation and penetration resulting from closure proximity surface ordinance detonation; and the design of a mobile Transportable Wastewater Advanced Refinement and Demonstration System. This included the design of state-of-the-art wastewater treatment equipment.

## **AFFILIATIONS**

American Society of Testing and Materials (ASTM) - Member

- Past Chairman of Liaison Committee
- Past Task Group Chairman for Compression of Geosynthetics

International Organization for Standardization (ISO) - Member

- Past U.S. delegate for the Hydraulic Properties of Geosynthetics

American Society of Civil Engineers (ASCE) - Member

- Past Faculty Advisor to Student Chapter at Georgia Tech  
Tau Beta Pi

International Geotextile Society (IGS) - Member

North American Geosynthetics Society (NAGS) - Member

Solid Waste Association of America (SWANA) - Member

National Society of Professional Engineers (NSPE) - Member

Florida Engineering Society (FES) - Member

**LIST OF PUBLICATIONS**

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- 85-1 Bonaparte, R., Williams, N.D., and Giroud, J.P., "Innovative Leachate Collection Systems for Hazardous Waste Containment Facilities", Proceedings of Geotechnical '85 Fabrics Conference, Cincinnati, Ohio, Jun 1985, pp. 9-34.
- 85-2 Houston, S.L., Houston, W.L., and Williams, N.D., "Thermo-Mechanical Behavior of Seafloor Sediments", Geotechnical Journal, ACE, III, No. II, Nov 1985, pp. 1249-1263.
- 86-1 Williams, N.D. and Grubert, P.A., "Performance Evaluation of Railroad Track Embankment Using Geotextiles", Geotechnical Engineering Report, Georgia Institute of Technology, Feb 1986.
- 86-2 Williams, N.D., and Houlihan, M., "Evaluation of Friction Coefficients between Geomembranes, Geotextiles, and Related Products", Proceedings of the Third International Conference on Geotextiles, Vol. III, Vienna, Austria, 1986, pp. 891-896.
- 86-3 Williams, N.D. and Papaioenou, G., "Evaluation of Geotextile Reinforced Embankments on the Farmer's Loop Road in Fairbanks, Alaska", Geotechnical Engineering Report, Georgia Institute of Technology, Jun 1986.
- 86-4 Bonaparte, R., Schmertmann, G.R., and Williams, N.D., "Seismic Design of Slopes Reinforced with Geogrids and Geotextiles", Proceedings of the Third International Conference on Geotextiles, Vienna, Austria, 1986, pp. 273-279.

- 87-1 Williams, N.D., Pohland, F.G., McGowan, K.C., and Saunders, F.M., Simulation of Leachate Generation from Municipal Solid Waste, USEPA, HWERL, Contract CR 8125 80010, Mar 1987.
- 87-2 Still, K.L. and Williams, N.D., "Engineering Properties of Stitch-Bonded Geotextiles", Geotechnical Engineering Report, Georgia Institute of Technology, Nov 1987.
- 87-3 Williams, N.D. and Houlihan, M.F., "Evaluation of Interface Friction Properties between Geosynthetics and Soils", Proceedings of Geosynthetics '87, Vol. II, New Orleans, LA 1987, pp. 616-627.
- 87-4 Williams, N.D. and Luna, J., "Selection of Geotextiles for Use with Synthetic Drainage Products", Geotextiles and Geomembranes, Elsevier Applied Science Publishers, Vol. 5, No. 1, 1987, pp. 45-61.
- 88-1 Frankenburger, P.C. and Williams, N.D., "Design of Reinforced Soil Structures Using Confined Tensile Strength", Vol. I, Geotechnical Engineering Report, Georgia Institute of Technology, Jan 1988.
- 88-2 Jang, D.J. and Williams, N.D., "Evaluation of Pullout and Creep Behavior of Geosynthetics in Soil by the X-Ray Technique", Vol. ii, Geotechnical Engineering Report, Georgia Institute of Technology, Jan 1988.
- 89-1 Luettich, S.M. and Williams, N.D., "Design of Vertical Drains Using the Hydraulic Conductivity Ratio Analysis", Proceedings of Geosynthetics '89, Vol. I., San Diego, CA, 1989, pp. 95-103.
- 89-2 Houlihan, M.F., Rogers, W.K., Willibey, G., and Williams, N.D., "Design and Construction of Synthetic-Grid Reinforced Embankment Over Soft Soils", Proceedings of Geosynthetics '89, Vol. I, San Diego, CA, 1989, pp. 148-159.
- 89-3 Langston, P.J. and Williams, N.D., "Design Methods for Reinforced Embankments on Soft Foundations", Proceedings of Geosynthetics '89, Vol. I, San Diego, CA, 1989, pp. 172-183.
- 90-1 Williams, N.D., Badu-Tweneboah, K., and Khatami, A., "GeoSynthetic Liner Systems", Proceedings of the 1990 Annual Meeting of ASCE South Florida Section, Miami, Florida, 14-15 Sept 1990.
- 90-2 Williams, N.D., and Badu-Tweneboah, K., "Design of Landfill Lining Systems on Sloping Waste Boundaries", Proceedings of the 1990 Annual Meeting of ASCE South Florida Section, Miami, Florida, 14-15 Sept 1990.



- 91-1 Williams, N.D., and Badu-Tweneboah, K., "Design Considerations for the Closure of Wastewater Treatment Sludge Landfills", Proceedings of 22nd Ohio River Valley Soils Seminar on Design and Construction with Geosynthetics, Lexington, Kentucky, 18 October 1991, pp. 10-1 to 10-15.
- 91-2 Weeks, N.D., and Williams, N.D., "Recovery of Fluids and Vapor in Fine-Grained Soils", Proceedings of Caribbean Haztech Environmental Conference & Exhibition, San Juan, Puerto Rico, November 12-14, 1991.
- 91-3 Badu-Tweneboah, K., Weeks, N.D., Williams, N.D., "Design of Lining Systems for Landfill Expansions", Proceedings of Caribbean Haztech Environmental Conference & Exhibition, San Juan, Puerto Rico, November 12-14, 1991.
- 92-1 Williams, N.D., Ojeshina, A.O., and Newman, S., "Waste Management Alternatives", Proceedings of the Lagos State Solid Waste Conference, Lagos, Nigeria, November, 1992.

**ROBERT C. BACHUS**

**geotechnical engineering,  
waste management,  
geosynthetics**

## **EDUCATION**

Stanford University: Ph.D., Geotechnical Engineering, 1982

University of Illinois at Chicago Circle: M.S., Civil Engineering, 1975

University of Illinois at Chicago Circle: B.S., Civil Engineering, 1974

## **PROFESSIONAL HISTORY**

GeoSyntec Consultants, Boca Raton, Florida, Senior Project Manager, 1990 - 1992; Associate, 1992 - 1994, Principal, 1994

Georgia Institute of Technology, Atlanta, Georgia, Assistant Professor of Civil Engineering, 1983 - 1990, Instructor, 1979 - 1983

Engineering Consulting Services provided to Soil Foundation Systems (Santa Clara, California), 1976 - 1979; Chattahoochee Geotechnical Consultants (Norcross, Georgia), 1975 - 1983.

## **REPRESENTATIVE EXPERIENCE**

### **Engineering Consulting**

Dr. Bachus has more than 15 years of experience in geotechnical engineering analysis, design, and testing. At GeoSyntec Consultants, Dr. Bachus has worked on several design/analysis teams performing stability analyses on landfill projects in Pennsylvania, New York, California, and Ohio. He is currently leading the permitting and design efforts for waste facilities in Florida and Illinois. He has also directed activities on the design and analysis of reinforced soil walls, design and permitting of vertical expansions of solid waste landfills, construction of solid waste landfills, stability of waste facilities constructed in areas underlain by karst geologic features and innovative gas and leachate management system designs. Dr. Bachus has worked on several remediation design projects, primarily on aspects related to slurry wall containment and soil/waste stabilization and compatibility testing. Dr. Bachus is responsible for technical oversight of GeoSyntec Consultants' Geomechanics and Environmental Laboratory in Norcross, Georgia. The laboratory specializes in conventional and specialized geotechnical testing, soil/geosynthetic interaction testing, and geo-environmental testing.

Prior to joining GeoSyntec Consultants, Dr. Bachus worked an independent geotechnical consultant responsible for providing testing and evaluation of laboratory and in situ test results on soft clays in New York and San Francisco and stiff soils in Atlanta and Virginia; analysis

of stability and design recommendations for earth dams; an assessment of potential causes and repair options of active landslides; an assessment of damage to structures due to blast-induced vibrations; recommendations for the use of lime and fly ash for highway construction throughout the southeastern U.S.; finite element analysis of the performance of geogrid-reinforced clay; and analysis of the stability of soft mine tailings and recommendations for stabilization.

### **Teaching**

Dr. Bachus taught in the geotechnical engineering program for 11 years at the Georgia Institute of Technology. He was primarily responsible for teaching graduate courses dealing with the engineering properties and physico-chemical properties of soils, lab and field testing techniques, seepage and slope stability, rock mechanics, soil construction, and soil/site improvement techniques in addition to undergraduate courses in soil mechanics and foundation engineering. He developed graduate courses in in situ testing, critical state soil mechanics, and numerical methods in geotechnical engineering, and co-authored a laboratory manual and spreadsheet tutorial for the undergraduate geotechnical program. He was recognized with four outstanding teaching awards from the American Society of Civil Engineers (ASCE) Student Chapter and a similar award from the Student Government Association.

While teaching at Georgia Tech, Dr. Bachus organized and prepared lectures for several continuing education short courses and technical seminars. Topics included: tieback and earth anchor design, soil reinforcement design, design and construction of stone columns, geosynthetic/soil interaction, advances in testing of geosynthetics, use of the pressuremeter in engineering practice, and recent developments in driven and pushed-in pressuremeters. He has also delivered several invited lectures to other universities and professional organizations on in situ testing techniques, design and use of geosynthetics, analysis of drilled shaft foundations, soil stabilization techniques, behavior of reinforced soil, and the engineering properties of residual soils. At GeoSyntec Consultants, Dr. Bachus has been an instructor for the Federal Highway Administration (FHWA)-sponsored course on design using geosynthetics and has taught university extension service short courses on the design, construction and testing of clay liners and on landfill design.

### **Research**

Dr. Bachus has been actively involved in research for the past 15 years. He is nationally-recognized for his expertise in the areas of soil stabilization, in situ testing, and by-product utilization, and maintains a wide range of geotechnical engineering research interests. While at Georgia Tech, he received financial support from several government agencies (National Science Foundation (NSF), FHWA, U.S. Environmental Protection Agency (USEPA), Georgia Department of Transportation (GaDOT)); electric utilities (Southern Company Services (SCS), Electric Power Research Institute (EPRI)); and several industrial sponsors. Soil stabilization and site foundation improvement interests include several studies on the design and use of

geosynthetics. This research focused on construction survivability of geotextiles, compression creep performance of drainage composites, effects of boundary conditions on geocomposite transmissivity, hydraulic conductivity ratio testing of geotextiles, performance of reinforced soils at high strain rates, stabilization of embankments constructed over soft subgrades, hydraulic and chemical transmission measurements through geomembranes, and clogging and blinding characteristics of geotextiles. Other foundation improvement work included the development of a design methodology for stone columns, laboratory scale modeling techniques of stone columns, an investigation which assessed the efficiency of deep dynamic densification of sands, and soil reinforcement techniques using both geosynthetic and steel mesh reinforcement. This latter work was directed at evaluating the pullout behavior of geogrids and steel mesh and developing a design manual for stability evaluation of reinforced soil walls.

Dr. Bachus has devoted a considerable effort in developing field testing equipment and analytical techniques for interpreting in situ test results. He is nationally recognized for his work on pressuremeter testing in stiff and cemented soils. These efforts have largely been directed towards the self-boring and pre-bored pressuremeter, but also include the piezoelectric cone penetrometer and the flat plate dilatometer. He has worked with this equipment in stiff cemented sands, soft to stiff clays, uncemented sands, residual soils and partially weathered rocks. To compliment the field testing activities, he maintains active interests in laboratory testing techniques and the comparison of laboratory to field results.

Dr. Bachus has worked on several projects which focused on the beneficial reuse of coal combustion by-products. These studies have considered bottom ash as a base material for highway construction, fly ash for subbase and subgrade stabilization, fly ash as a filler in asphalt concrete, and coal ash as structural fill. At GeoSyntec Consultants, he is currently developing an index system in collaboration with SCS and EPRI for characterizing coal combustion by-products.

At GeoSyntec Consultants, Dr. Bachus was recently the Project Manager of a comprehensive research project for the US Air Force which considered the performance of geosynthetic reinforced soil walls subjected to blast loading. This work utilized specially designed laboratory testing equipment to simulate blast loading, centrifuge modeling and dynamic three-dimensional finite element modeling of the reinforced soil system.

Dr. Bachus has authored or co-authored more than 50 technical papers and reports. He recently was co-editor of the ASTM publication STP 1084, *Deep Foundation Improvements: Design, Construction and Testing*.

## **AFFILIATIONS**

International Geotextile Society  
North American Geosynthetics Society  
American Society for Testing and Materials  
    D-18 Geotechnical Committee  
    D-35 Geosynthetics Committee  
American Society of Civil Engineers  
    Geotechnical Committee, Georgia Section  
    Soil Properties Committee, Member  
International Society of Soil Mechanics and Foundation Engineering  
Transportation Research Board  
    Soil Properties Committee (A2LO2), Member (Friend of the Committee)  
Technical Affiliate, Association of Drilled Shaft Contractors

**LIST OF PUBLICATIONS**

**R.C. BACHUS**

- 75-1 Bachus, R.C., Silver, M.L., and Priemer, R., *"Noise Assessment of the Chicago Transit Authority Rail Rapid Transit System"*, Report UMTA-IL-11-007-2, Dept. of Transportation, Urban Mass Transit Authority, Jul 1975.
- 75-2 Bachus, R.C., *"Review of Filter Design Criteria for Clay"*, Stanford University Research Report submitted to Dr. J.L. Sherard, Dec 1975.
- 76-1 Bachus, R.C. and Clough, G.W., *"An Evaluation of the Technical Feasibility of a Slurry Trench Cut-Off for the Excavation for the Tensas-Cocodrie Pumping Plant and Review of Slurry Trench Specifications"*, report submitted to Vicksburg district, U.S. Army Corps of Engineers, 1976.
- 78-1 Bachus, R.C. and Clough, G.W., *"Self-Boring Pressuremeter Testing of San Francisco Bay Mud for Muni Track Extension"*, report submitted to Woodward-Clyde Consultants, San Francisco, Sep 1978.
- 79-1 Bachus, R.C., and Clough, G.W., *"Self-Boring Pressuremeter Testing of Hudson River Soils for the Westside Highway Project, New York City"*, report submitted to DOT, State of New York, Albany, Aug 1979.
- 80-1 Bachus, R.C., Sitar, N., and Clough, G.W., *"Behavior of Weakly Cemented Soil Slopes under Static and Seismic Loading Conditions"*, Report No. 44, The John A. Blume Earthquake Engineering Center, Stanford University, Jun 1980.
- 81-1 Bachus, R.C., Clough, G.W., Sitar, N., Shafii-Rad, N., *"Cemented Sands under Static Loading"*, Journal of the Geotechnical Engineering Division, ASCE, Vol. 107, No. GT6, Jun 1981.
- 81-2 Bachus, R.C., Clough, G.W., Sitar, N., Shafii-Rad, N., Crosby, J., and Kaboli, P., *"Behavior of Weakly Cemented Soil Slopes under Static and Seismic Loading Conditions"*, Vol. II, John A. Blume Earthquake Engineering Center report, Stanford University, Jul 1981.
- 82-1 Bachus, R.C. and Clough, G.W., *"An Investigation of Sampling Disturbance in Weakly Cemented Sand"*, Engineering Foundation Conference on Updating Subsurface Sampling and In-Situ Testing, Santa Barbara, CA, Jan 1982.
- 82-2 Bachus, R.C. and Mitchell, J.K., *"In-Situ Soil Testing - Part A - Session Reporters Summary"*, Engineering Foundation Conference on Updating Subsurface Sampling and In-Situ Testing, Santa Barbara, CA, Jan 1982.

- 82-3 Bachus, R.C., Barksdale, R.D., and Calnan, M.B., "*Settlements of a Tower on Residual Soil*", Proceedings, ASCE Specialty Conference on Engineering and Construction in Tropical Residual Soils, Honolulu, HI, Jan 1982.
- 83-1 Bachus, R.C. and Barksdale, R.D., "*Design and Construction of Stone Columns*", Report No. FHWA/RD-83/026, Dec 1983.
- 84-1 Bachus, R.C. and Pohland, F.G., "*Critical Review and Summary of Leachate and Gas Production from Landfills*", Final Report to U.S. Environmental Protection Agency, Cooperative Agreement No. CR809997, Municipal Environmental Research Laboratory, Cincinnati, OH, Mar 1984.
- 84-2 Bachus, R.C. and Barksdale, R.D., "*Vertical and Lateral Behavior of Model Stone Columns*", Proceedings, International Conference on In-Situ Soil and Rock Reinforcement, Paris, Oct 1984.
- 85-1 Bachus, R. C. and Pohland, F.G., "*Critical Review and Summary of Analytical Methods for the Determination of the Hydraulic Integrity of Synthetic Liners*", Final Report, Cooperative Agreement No. CR810807, U.S. Environmental Protection Agency, Hazardous Waste Research Laboratory, Cincinnati, OH, Apr 1985.
- 85-2 Bachus, R.C., "*The Effects of Sample Disturbance on the Stress-Deformation Behavior of Soft Sandstone*", Proceedings, 36th Annual Highway Geology Symposium, Clarksville, IN, May 1985.
- 85-3 Bachus, R.C., "*The Use of the Pressuremeter to Evaluate the Strength-Deformation Characteristics of Soft Rocks*", Proceedings, 26th U.S. Symposium on Rock Mechanics, Rapid City, SD, Jun 1985.
- 85-4 Bachus, R.C., "*In-Situ Testing to Evaluate the Deformation Characteristics of Residual Soils*", Invited Discussion, In-Situ Testing Techniques Session Report, XI International Conference on Soil Mechanics and Foundation Engineering, San Francisco, CA, Aug 1985.
- 86-1 Bachus, R.C. and Hughes, J.M.O., "*Feasibility of the Development of an Instrument to Measure the In-Situ Stress and pore Pressure in Soils Below 6000 Meters in Depth*", Summary Research Report Submitted to The Canadian Geotechnical Survey, May 1986.
- 86-2 Bachus, R.C., Hughes, J.M.O., Benoit, J., and Deshpande, S.C., "*The Pressuremeter: An Insitu Testing Instrument Which Provides Useful Data for Geotechnical Design*", prepared for "How-To" Session on Pressuremeter Testing,



ASCE Specialty Conference, Use of Insitu Tests in Geotechnical Engineering, VPI, Jun 1986.

- 86-3 Bachus, R.C., "*The Pressuremeter Test and Its Role in Evaluating the Engineering Behavior of Soft Rock*", 8th Danube-European Conference on Soil Mechanics and Foundation Engineering, Nüremberg, Federal Republic of Germany, Sep 1986.
- 87-1 Bachus, R.C., Benoit, J., and Hughes, J.M.O., "*The Use of In-Situ Soil Tests To Evaluate the Engineering Properties of Stiff Soils*", 65th Transportation Research Board Annual Meeting, Session on Properties of Overconsolidated and Stiff Clay Soils and Shales, Jan 1987.
- 87-2 Bachus, R.C., "*Lesson Learned from European Practice on the Use of Stone Columns for Site Improvement*", 38th Annual Highway Geology Symposium, Pittsburgh, PA, May 1987.
- 87-3 Bachus, R.C., Benoit, J., and Hughes, J.M.O., "*The Role of Pressuremeter Testing in Geotechnical Exploration Programs*", Journal of The Boston Society of Civil Engineers, Jun 1987.
- 87-4 Bachus, R.C. and Collins, S., "*The Use of Hypoelasticity to Model the Behavior of Sands*", International-Workshop on Constitutive Equations for Granular Non-Cohesive Soils, Case Western Reserve University, Jul 1987.
- 87-5 Bachus, R.C., "*Earth Reinforcement: Backfill Interaction and Behavior*", Southeastern Transportation and Geotechnical Engineering Conference, Hot Springs, AK, Oct 1987.
- 88-1 Bachus, R.C., "*Large Scale Pullout Resistance of Geogrid Reinforcement*", 66th Annual Meeting of the Transportation Research Board, Session on Effects of Geosynthetics on Soil Properties, Jan 1988.
- 88-2 Bachus, R.C. and Mayne, P., "*Profiling OCR in Clays by Piezocone Soundings*", 1st International Symposium on Penetration Testing (ISOPT1), Mar 1988.
- 88-3 Bachus, R.C. and Briaud, J.L., "*Full Displacement and Driven Pressuremeter Testing of Soils*", Specialty Session, 1st International Symposium on Penetration Testing (ISOPT1), Mar 1988.
- 88-4 Bachus, R.C. and Ospina, R., "*Pullout Resistance of the Georgia Stabilized Earth Mesh Embedded in Compacted Sand*", Georgia Department of Transportation, Office of Materials and Research, Aug 1988.

- 88-5 Bachus, R.C. and Deh-Jang, D., "*Evaluation of the Long Term Compression Creep Response of Geocomposite Drainage Cores*", Research Report submitted to Monsanto Chemical Company, Aug 1988.
- 88-6 Bachus, R.C. and Larrimore, C.L., "*Ash Utilization in Highway Construction-Georgia Demonstration Project*", EPRI Report, Oct 1988.
- 89-1 Bachus, R.C., "*Radiographic Monitoring of Laboratory Scale Model Tests: Its Role in the Interpretation of Geotechnical Test Results with Emphasis on the Advanced Study of the Pressuremeter*", Final Project report submitted to NSF, Mar 1989.
- 89-2 Bachus, R.C. and Larrimore, C.L., "*Ash Field Demonstration Project - Mississippi*", Research report submitted to Southern Company Services, Inc., Research and Development Department, May 1989.
- 89-3 Bachus, R.C. and Barksdale, R.D., "*Design Methodology for Foundations on Stone Columns*", 1989 Foundation Engineering Congress, Northwestern University, Jun 1989.
- 89-4 Bachus, R.C. and Mayne, P.W., "*Penetration Pore Pressures by CPTU, DMT and SBP*", Proceedings, 12th ICSMFE, Rio de Janeiro, Aug 1989.
- 89-5 Bachus, R.C., "*Use of Coal Ash in Florida Highway Construction*", Research Report submitted to Southern Company Services, Inc., Florida DOT and Gulf Power Company, Aug 1989.
- 89-6 Bachus, R.C. and Gallup, R.A., "*Hydraulic Performance of Geocomposite Drainage Products*", Research Report submitted to Contech Construction Products, Sept 1989.
- 89-7 Bachus, R.C., "*Physical and Engineering Properties of Coal Ash*", Design and Construction Applications Using Ash: A Technical Workshop, Atlanta, Georgia, Nov 1989.
- 89-8 Bachus, R.C., "*Soil Amendment and Subgrade Stabilization*", Design and Construction Application Using Ash: A Technical Workshop, Atlanta, Georgia, Nov 1989.
- 90-1 Bachus, R.C., "*Use of Plant Daniel Fly Ash as Mineral Filler in Asphalt Concrete*", Research Report submitted to Southern Company Services, Mar 1990.
- 90-2 Bachus, R.C. and Clough, G.W., "*Self-Boring Pressuremeter Tests in Weakly Cemented Sands*", Accepted for Publication, ASCE Geotechnical Engineering Journal, Sept 1990.

- 90-3 Bachus, R.C. and Yalaza, D., "*Development of Design Methodology for Mechanically Stabilized Earth Walls*", Research Report submitted to Beazer West, Aug 1990.
- 90-4 Bachus, R.C., "*Deep Foundation Improvement Techniques-Current State of Practice*", Chairman's Report, ASTM Special Technical Publication STP 1084, Sept 1990.
- 90-5 Bachus, R.C., "*Behavior of Drilled Shaft Foundations in Residual Soil Partially Weathered Rock*", Workshop on Drilled Shaft Foundations, Association of Drilled Shaft Contractors, Atlanta, GA, Sep 1990.
- 90-6 Bachus, R.C., Stone, R.C., and Fiest, P.W., "*Performance of Drilled Shaft Foundation for LLWAS Structures*", Research Report submitted to Federal Aviation Administration, Southern Region, Oct 1990.
- 90-7 Bachus, R.C. and Narejo, D., "*Construction Survivability of Woven and Non-Woven Geotextiles*", Research report submitted to Exxon Chemical Company, Oct 1990.
- 91-1 Bachus, R.C. and Larrimore, C.L., "*Lesson Learned from the Use of Coal Ash in Highway Construction*", Accepted for Publication Transportation Research Board, Jan 1991.
- 91-2 Bachus, R.C. and Larrimore, C.L., "*Use of Coal Ash in Highway Construction*", International Symposium in Ash Utilization, Shanghai, China, Mar 1991.
- 91-3 Bachus, R.C. and Larrimore, C.L., "*Index System as a Means of Coal Ash Classification*", International Symposium on Ash Utilization, Shanghai, China, Mar 1991.
- 91-4 Bachus, R.C., "*Use of High Volume Fly Ash Concrete - An Industry Survey*", Workshop on High Volume Fly Ash Concrete, Southern Company Services, Atlanta, GA, May 1991.
- 91-5 Bachus, R.C. and Larrimore, C.L., "*Development of Coal Ash Index System for Bottom Ash*", ASCE Power Engineering Division Specialty Conference, Aug 1991.
- 92-1 Bachus, R.C. and Swan, R.H., "*Shear Strength of Geosynthetic Clay Liners*", presented at the Geosynthetic Clay Liner Workshop, Environmental Protection Agency, Cincinnati, OH, Jul 1992.

- 92-2 Bachus, R.C. and Swan, R.H., "*Shear Strength of Geosynthetic Clay Liners*", presented at Geosynthetic Clay Liner Roundtable Discussion, Browning-Ferris Industries, Memphis, TN, Sep 1992.
- 93-1 Bachus, R.C., Reid, R.A., Olen, K.L., and Frigaszy, R., "Response of Geogrid-Reinforced Soil Subjected to Blast Loading", *Proceedings of the 6th International Symposium on Interaction of Nonnuclear Munitions with Structures*, Panama City, FL, May 1993.
- 93-2 Bachus, R.C., Soderman, K.L., and Swan, R.H., "*Factors which Affect Soil/Geosynthetic and Geosynthetic/Geosynthetic Interface Shear Strength for Materials Used in Landfill Lining Systems*", American Society of Civil Engineers, 1993 Annual Meeting, Naples, FL, Oct 1993.
- 93-3 Bachus, R.C., Giroud, J.P., and Darrasse, J., "Hyperbolic Expression for Soil-Geosynthetic or Geosynthetic-Geosynthetic Interface Shear Strength", *Geotextiles and Geomembranes*, Elsevier Science Publishers, Vol. 12, Dec 1993, pp. 275-286.
- 94-1 Bachus, R.C., Schmertmann, G.R., and Swan, R.H., "Shear Strength of Geosynthetic Clay Liners", *Proceedings of the Geosynthetic Clay Liner Symposium*, Nüremberg, Germany, Apr 1994.

**NANDRA D. WEEKS, P.E.**

**remedial design,  
environmental assessments,  
contaminant transport analyses,  
project management**

## **EDUCATION**

Nova University: M.B.A, 1990

Georgia Institute of Technology: Graduate Studies in Geotechnical Engineering, 1987

Georgia Institute of Technology: B.S., Civil Engineering, 1986

## **PROFESSIONAL ENGINEER REGISTRATION**

Florida, P.E. Number 45428

## **PROFESSIONAL HISTORY**

GeoSyntec Consultants, Boca Raton, Florida, Project Engineer, 1990 - date

Southern Bell Telephone Company, Orlando, Florida, 1988-1990

Southern Bell Telephone Company, Marietta, Georgia, 1987-1988

Research Assistant, Georgia Institute of Technology, 1986-1987

Law Environmental Inc., Kennesaw, Georgia, 1986-1987

Southern Bell Telephone Company, Jacksonville, Florida, 1983-1985

## **REPRESENTATIVE EXPERIENCE**

Since joining GeoSyntec Consultants, Ms. Weeks has been the Project Manager and/or Project Engineer for environmental assessments, remedial investigations, and feasibility studies, and has provided remedial design for several projects located in South Florida, Virginia, New Jersey, Philadelphia, and South Carolina. She has managed field activities including drilling and monitoring well installation, ground-water, soil gas, and soil sampling, soil excavation, well-point systems, and geophysical testing. Investigatory/remedial activities have included source removal, management of migration, and design of pump and treat systems. The pump and treat systems have included recovery of both pure phase liquids and contaminated ground water. Ms. Weeks has also been involved in developing site-specific three-dimensional ground-water flow and contaminant transport models. These models have been used for source discovery, expert witness testimony, cost allocation, predictive behavior, and remedial design.

Ms. Weeks has prepared and reviewed Site Management Plans, Remedial Design Workplans, Contamination Assessment Reports, Remedial Action Plans, and Resource Conservation and Recovery Act (RCRA) Facility Investigation Reports. She has negotiated with the U.S. Environmental Protection Agency (USEPA) to develop a Statement of Work for a Superfund Site and has designed RCRA Facility Investigations. Ms. Weeks is familiar with environmental regulations and has demonstrated the ability to work well with local, state, and federal (USEPA) regulatory officials. Her experience includes project management of several sites under Administrative Order.

Ms. Weeks has played a major role in developing the Corporate Health and Safety Program Plan and in addition, has written and/or reviewed site-specific health and safety plans for several sites, including a Superfund site in New Jersey.

Ms. Weeks has also performed modeling and site assessment activities for several service stations in Puerto Rico. These assessments included analysis of remedial technologies and design of ground-water and product recovery systems.

As a Research Assistant at the Georgia Institute of Technology, Ms. Weeks was involved with two research projects: (i) development of a design manual and technical notes for geotextile reinforced embankments over soft foundations; and (ii) numerical evaluation of geotextile reinforced embankments on Farmers Loop Road - Fairbanks, Alaska.

The first project included formulation of a design manual which summarized analytical requirements and case histories for design of embankments constructed over weak foundations. The second project involved the utilization of numerical analysis to evaluate the potential impact of a geotextile, base reinforced embankment on Farmers Loop Road in Fairbanks, Alaska. The site was complicated by a subgrade which was subjected to freeze/thaw cycles.

Ms. Weeks also has significant experience in design of geosynthetics. She has worked on numerous landfill design projects involving geomembrane liners, geogrid reinforcing layers, high strength geotextiles, geocomposite drainage layers, and embankments over soft subgrades.

#### **AFFILIATIONS**

American Society of Civil Engineers  
Florida Engineering Society  
Chi Epsilon  
International Geotextile Society  
North American Geosynthetics Society

\* \* \* \* \*

## PUBLICATIONS

- 91-1 Weeks, N.D., and Williams, N.D., "Recovery of Fluids and Vapor in Fine-Grained Soils", Proceedings of Caribbean Haztech Environmental Conference & Exhibition, San Juan, Puerto Rico, 1991.
- 91-2 Badu-Tweneboah, K., Weeks, N.D., Williams, N.D., "Design of Lining Systems for Landfill Expansions", Proceedings of Caribbean Haztech Environmental Conference & Exhibition, San Juan, Puerto Rico, 1991.

**PAUL M. STOUT**

**hydrogeology,  
geochemistry,  
environmental assessment,  
marine science**

## **EDUCATION**

University of California, San Diego,: Ph.D., Earth Sciences, 1985

Duke University: M.S., Geology, 1979

Colgate University: A.B., Geology, 1977

## **PROFESSIONAL HISTORY**

GeoSyntec Consultants, Boca Raton, Florida, Senior Project Hydrogeologist, 1992-present  
ERM-South, Inc., Boca Raton, Florida, Project Hydrogeologist, 1990-1992; Tampa, Florida,  
Project Hydrogeologist, 1989-1990

Geos, Inc., Tampa, Florida, Laboratory Director, 1990

East Carolina University, Greenville, North Carolina, Institute of Coastal and Marine  
Resources, Assistant Scientist; Geology, Adjunct Assistant Professor, 1988-1989

University of California, Santa Barbara, California, Marine Science Institute, Postgraduate  
Research Geochemist, 1986-1988

University of Miami, Florida, Rosenstiel School of Marine and Atmospheric Sciences,  
Postdoctoral Associate, 1985-1986

Scripps Institution of Oceanography, La Jolla, California, Graduate Research and Teaching  
Assistant, 1979-1984

U.S. Geological Survey, San Juan, Puerto Rico, Geologist, 1978

## **REPRESENTATIVE EXPERIENCE**

Dr. Stout has more than 15 years of technical and managerial experience working with universities, government, and private industry. His consulting experience includes serving as hydrogeologist for the development of landfill sites in Florida, Illinois and Tennessee. He has performed numerous contamination assessments involving petroleum hydrocarbons, heavy metals, PCBs and pesticides at multiple sites throughout Florida and the southern U.S. He has been responsible for designing and implementing all phases of surface and subsurface investigations. Work on these projects included evaluation of the interactions, transport and fate of solutes and contaminants in surface-water and ground-water environments. Dr. Stout has also testified as an expert witness for a legal case involving contaminant fate and transport associated with former manufactured gas facilities.



Dr. Stout has served as director of a state-certified (Florida) analytical laboratory specializing in analyses of environmental samples and drinking water. He was responsible for overall operation of the laboratory, and also served as gas chromatography analyst for volatile organic compounds and environmental microbiologist.

While at East Carolina University, Dr. Stout served as principal investigator for an EPA sponsored study of heavy metal pollution in rivers and estuaries of North Carolina. The work included collection and analysis of sediment and water samples to evaluate the impacts from both point and non-point metal sources. He was also awarded research grants from the National Science Foundation and North Carolina Board of Science and Technology to investigate the fate and transport of metals in coastal and estuarine environments.

During his tenure as a research geochemist at the University of California, Santa Barbara, Dr. Stout developed new analytical techniques to measure trace metals in natural waters and sediments. The techniques were used to conduct real-time analyses while in the field to investigate the behavior and distribution of cobalt and copper in aerobic and anaerobic environments. He also participated in an assessment of the impacts from a freighter that sank off the central California coast, while transporting more than 20,000 tons of copper ore.

While employed by the University of Miami, Dr. Stout conducted research on the hydrogeology and hydrogeochemistry of the Biscayne and other aquifers in South Florida. The research included examining core borings from more than 50 deep wells, analyzing the organic and inorganic chemical composition of ground water and rocks, and modeling geochemical processes affecting water quality of the underlying aquifers. He also taught a graduate course on hydrogeology and ground water, emphasizing both physical and chemical processes, as well as applied aspects of the field.

During his affiliation with the Scripps Institution of Oceanography, Dr. Stout performed geochemical research investigating water-rock interactions in calcium carbonate and siliceous sedimentary environments. These studies included determining quantities and rates of chemical reactions at low and moderate temperatures. He developed fluid transport/reaction models to describe mineral transformation processes and their potential effects on acoustic properties of the sedimentary formations. Dr. Stout also served as a staff scientist aboard the D/V Glomar Challenger during the Leg 85 expedition of the Deep-Sea Drilling Project (DSDP).

Dr. Stout has also conducted investigations of sedimentary processes in coastal and nearshore marine environments. Part of this work occurred while working for the U.S. Geological Survey in San Juan, Puerto Rico. Additional studies were performed on the Atlantic coastal margin in New Jersey, Florida and North Carolina.

## **AFFILIATIONS**

Association of Ground Water Scientists and Engineers  
American Geophysical Union  
American Chemical Society  
Sigma Xi, the Scientific Research Society  
Southeast Florida Geological Society

## **PROFESSIONAL REGISTRATION**

Florida, Professional Geologist #1118  
North Carolina, Licensed Geologist #1064

**LIST OF PUBLICATIONS**

**P.M. STOUT**

- 77-1 Stout, P.M. and McClennen, C.E., "*Buried valley segments on the continental shelf off Delaware Bay and New Jersey: new data and alternative reinterpretations*", GSA Abstracts with Programs, 9, no. 3, 1977, p. 322.
- 79-1 Stout, P.M., "*Calcium carbonate sedimentation on the northeast insular shelf of Puerto Rico*", M.S. thesis, Duke University, 1979, 107 p. .
- 79-1 Stout, P.M., Chen, Y.T. and Pilkey, O.H., "*The role of submarine canyons in construction of the U.S. Mid-Atlantic Continental Rise*", GSA Abstracts with Programs, 11, no. 7, 1979, p. 525.
- 82-1 Stout, P.M., "*Hydrothermal alteration of sediments, Guaymas Basin, Gulf of California*", Inter. Assoc. Sediment. Congress, 11, 1982, p. 46.
- 83-1 Stout, P.M., and Campbell, A.C., "*Hydrothermal alteration of near-surface sediments, Guaymas Basin, Gulf of California*", In Larue, D.K. and Steel, R.J. eds., *Cenozoic Marine Sedimentation, Pacific Margin, USA*. Pacific Section SEPM, 1983, pp. 223-231.
- 83-2 Stout, P.M., Kastner, M. and Gieskes, J.M., "*Diagenesis of calcareous sediments of the eastern equatorial Pacific: Sr/Ca variations*", GSA Abstracts with Programs, 15, no. 6, 1983, p. 699.
- 83-3 Stout, P.M., "Diagenesis of biogenic sediments from the eastern equatorial Pacific, DSDP Leg 85". EOS, 64, no. 45, 1983, p. 733.
- 84-1 Lesmes, D., Gieskes, J.M., Campbell, A.C., Stout, P.M., and Brumsack, H.J., "*Geochemistry of hydrothermally altered surface sediments in the Guaymas Basin - Gulf of California*," EOS, 65, no. 45, 1984, p. 974.
- 85-1 Stout, P.M., "*Interstitial water chemistry and diagenesis of biogenic sediments from the equatorial Pacific, DSDP Leg 85*", Init. Repts. DSDP, 85, 1985, pp. 805-820.
- 85-2 Stout, P.M., and Kastner, M., "*Extent of calcite recrystallization in deep-sea sediments: Sr/Ca distributions and oxygen isotopes*", GSA Abstracts with Programs, 17, no. 7, 1985, p. 729.
- 85-3 Stout, P.M., "*Chemical diagenesis of pelagic biogenic sediments from the equatorial Pacific*", Ph.D. dissertation, University of California, San Diego, 221 p.
- 87-1 Stout, P.M. Johnson, K.S. and Sakamoto-Arnold, C.M., "*Cobalt distributions in coastal and pelagic regions of the Pacific Ocean*", EOS, 68, 1987, p. 1754.

- 88-1 Johnson, K.S., Stout, P.M. and Sakamoto-Arnold, C.M., "*Cobalt and copper distributions in the waters of Santa Monica Basin, California*", *Nature*, 332, 1988, pp. 527-530.
- 88-2 Stout, P.M., Johnson, K.S. and Sakamoto-Arnold, C.M., "*Shipboard determination of copper in seawater using flow injection analysis with chemiluminescence detection*", *EOS*, 69, 1988, p. 1264.
- 89-1 Stout, P.M., Riggs, S.R., Powers, E.R., Bray, J.T., Moore, R., and Hamilton, J.C., "*Concentration and distribution of heavy metal pollutants in organic-rich muds of the Pamlico River Estuarine System*", *GSA Abstracts with Programs*, Vol. 21, No.3, 1989.
- 89-2 Riggs, S.R., Powers, E.R., Bray, J.T., Stout, P.M. et al., "*Heavy metal pollutants in organic-rich muds of the Pamlico River Estuarine System: their concentration, distribution, and effects upon benthic environments and water quality*", *USEPA-APES Final Report: The Pamlico River*, 1989, 190 p.
- 91-1 Baker, P.A., Stout, P.M., Kastner, M., and Elderfield, H., "*Large-scale advection of seawater through oceanic crust in the central equatorial Pacific*" *Earth Planet. Sci. Lett.*, v. 105, 1991, p. 522-533.

**JOHN H. RAYMER, P.G.**

**environmental site characterization  
karst hydrogeology  
subsurface stratigraphy  
field geology & drilling  
remedial construction management**

## **EDUCATION**

University of Oklahoma: M.S. Geology, 1987

Furman University: B.S. Geology, 1981

## **PROFESSIONAL REGISTRATION**

Georgia, Professional Geologist #828

## **PROFESSIONAL HISTORY**

GeoSyntec Consultants (formerly GeoServices Inc. Consulting Engineers), Norcross,  
Georgia, Senior Staff Geologist, 1991-date

Dunn Geoscience Corporation, Atlanta, Georgia, Staff Geologist, 1989-1991

ATEC Environmental Consultants, Marietta, Georgia, 1988-1989

Contract Geologist, Norman, Oklahoma, 1987

Oklahoma Geological Survey, Norman, Oklahoma, Research Assistant, 1985-1987

University of Oklahoma, Norman, Oklahoma, Graduate Teaching Assistant, 1981-1984

## **REPRESENTATIVE EXPERIENCE**

Mr. Raymer has over six years of experience characterizing subsurface geology and hydrogeology in association with environmental and petroleum investigations. He specializes in surface and subsurface stratigraphy, and in designing and leading drilling programs.

At present, Mr. Raymer is the site geologist for a large RI/FS project in the Maryland Coastal Plain. His work at that site has included the identification and three-dimensional delineation of contaminant plumes using the hydropunch sampling system in combination with other sampling techniques. With the hydropunch sampling technique, Mr. Raymer was able to show conclusively and economically the pathway by which leachate was migrating from the previously constructed, unlined disposal cells, around an intermittent underlying clay layer, and into the aquifer below. These critical zones of flow, which Mr. Raymer delineated with hydropunch sampling of the ground water, will now be used as the focus for an economical remediation system based on a concise understanding of the site hydrogeology.

Mr. Raymer recently investigated ground-water contamination in a karst (cavernous limestone) hydrogeologic environment. The site contained very high concentrations of both light and dense volatile organic solvents. Mr. Raymer showed that contaminant migration and storage was controlled by the hydrostratigraphy and by the pore-system architecture in the rock and soil. To complete the analysis, Mr. Raymer examined outcrops and air-photographs, mapped containment dissolution channel patterns using rock-probing techniques, mapped adsorption trails through the overburden using gas chromatograph analysis of soil samples, analyzed rock cores, and installed and sampled ground-water monitoring wells.

Mr. Raymer has been involved with tracking mercury that has been leaching into the ground water from waste lagoons on a RCRA site on the Georgia Coast. His principal role has been the design and installation of a monitoring well network in a tidal salt marsh. By applying his knowledge of drilling, hydrogeology, and subsurface stratigraphy, he was able to develop and implement a low-cost method of installing wells in a heaving sand formation while maintaining hydraulic separation between the sand and overlying marsh muck, in an area inaccessible to normal drilling equipment.

Mr. Raymer has managed over twenty environmental, hydrogeological, hazardous waste investigations. He has supervised the installation of about 200 environmental test wells, including several that involved over 350 ft (105 m) of continuous coring and borehole geophysics. He has also conducted environmental and geotechnical mapping on several sites, including a 4000 acre (1600 hectare) tract of old mining property. Mr. Raymer also recently performed construction surveillance on a major remediation project that involved excavation, transport, and off-site disposal of hazardous wastes.

Mr. Raymer worked on several projects for the Oklahoma Geological Survey. The two most significant projects concerned the subsurface geology and petroleum potential of northwestern Oklahoma; and the oil and gas potential of Permian strata in the southern Mid-Continent. In the first project, Mr. Raymer correlated over 4,000 well logs, developed cross-sections, mapped the subsurface structure and stratigraphy, and analyzed production trends in terms of facies controls. In the second project, Mr. Raymer mapped production, compiled statistics, and analyzed pressure decline for Permian oil and gas reservoirs in Oklahoma, Kansas and the Texas Panhandle.

Mr. Raymer's M.S. thesis was entitled "*Lithostratigraphy and Depositional Environments of the Deese Group (Middle Pennsylvanian) of the Ardmore Basin, Southern Oklahoma*". In this study, he measured over 60,000 ft (18,300 m) of stratigraphic sections in a 7,000-ft (2100-m) sequence of interbedded sandstone and shale that was deposited in a tectonically active environment.

## **AFFILIATIONS**

Geological Society of America  
Georgia Geologic Society  
Georgia Ground-Water Association



**PUBLICATIONS OF**  
**JOHN H. RAYMER**

"Habitat of Petroleum in Permian Rocks of the Mid-continent Region", J.A. Campbell and others, in Permian Rocks of the Mid-Continent Region: Society of Economic Paleontologists and Mineralogists, Special Publication No. 1, 1988.